

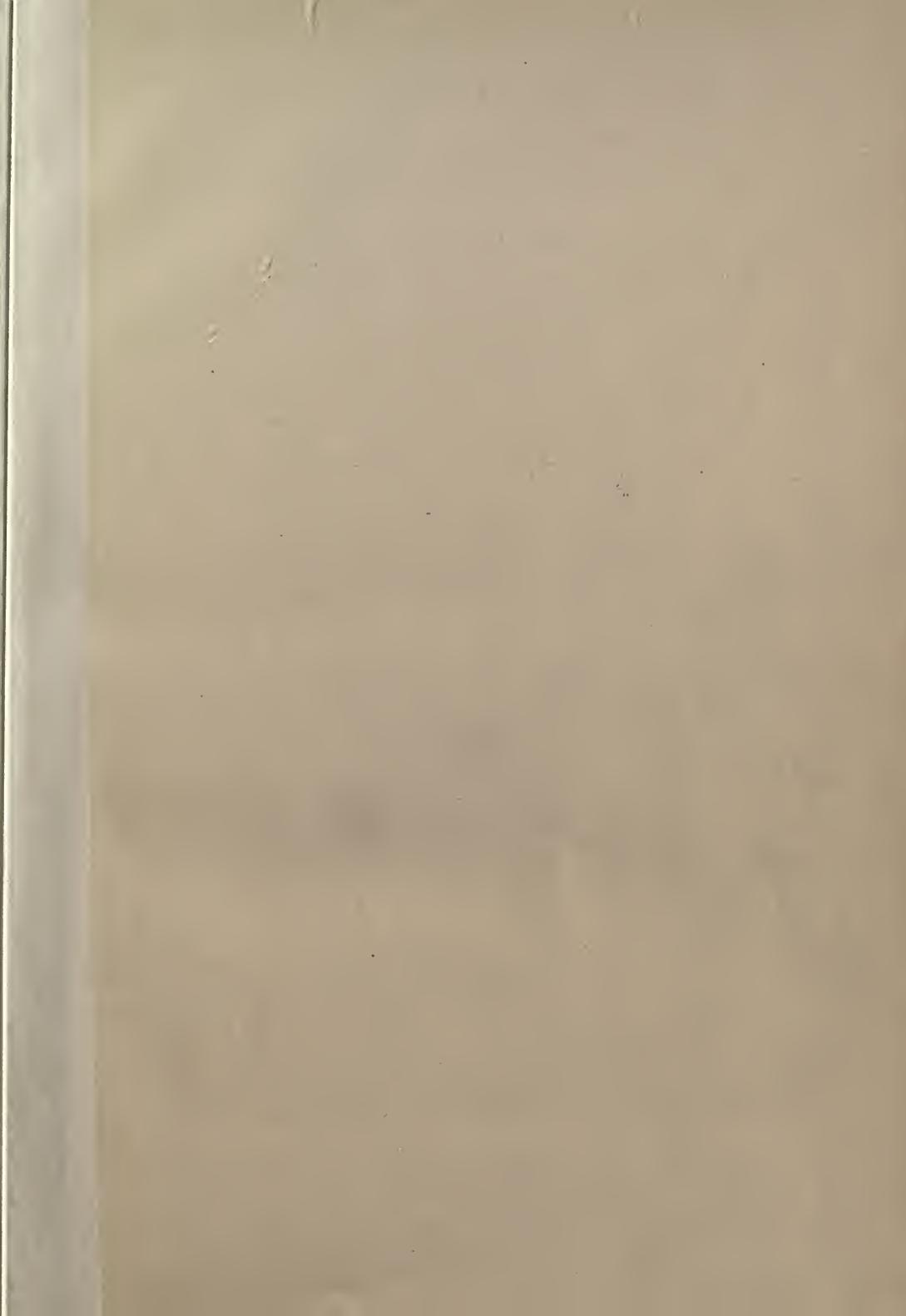
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# THE DESIGN AND EQUIPMENT OF SMALL CHEMICAL LABORATORIES

BY

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## PREFACE.

It very often happens that the young chemist, perhaps fresh from college, is called upon to design and equip a laboratory when his knowledge of how it is to be done is rather meagre. The experienced chemist, confronted with the task, can usually bring to bear upon the subject sufficient practical knowledge, gained from other laboratories in which he has worked, to arrange and equip a laboratory which will meet fully his requirements; and can do so, in many instances, better than any one else can do it for him. The inexperienced college graduate, on the other hand, has only a knowledge, in most cases, of the laboratory of the college where he graduated. Many things there are done by means which he cannot command in his new position—for instance, the ventilation of the hoods and rooms is perhaps controlled by mechanical draft, when in his new quarters he will not have the power to run fans; or the gas supply may be that of the city, when his new laboratory will be located at the mines. To these young chemists, the following suggestions on the design and equipment of small laboratories are made, but it is also hoped that even experienced chemists will find in them some points of use and help.

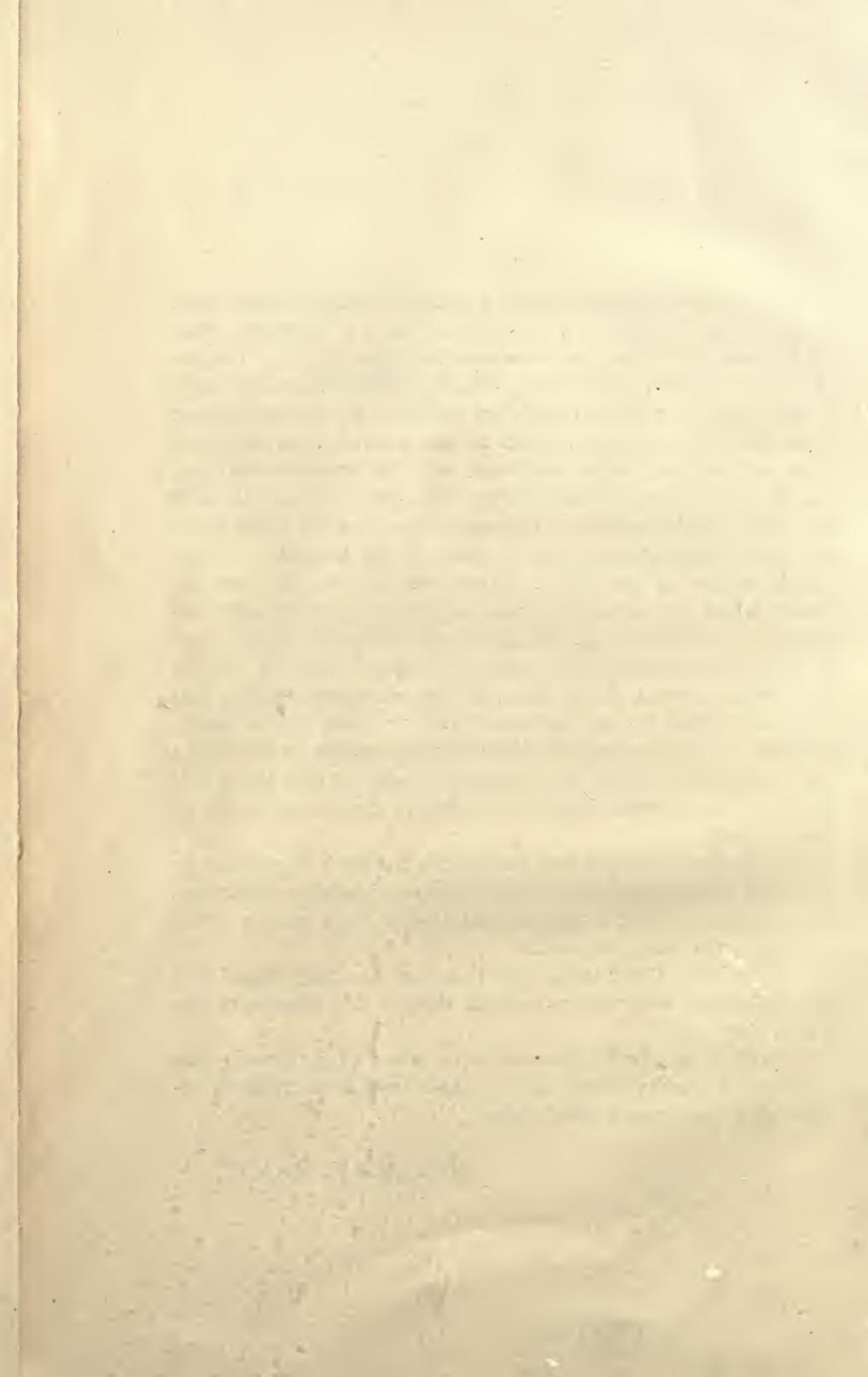
It has been thought best throughout the book to mention by name the firms making the various forms of apparatus described. This is done in order to enable the reader to more readily obtain the appliances which he needs.

We wish to thank those manufacturers who have loaned cuts for illustrating the various special devices for laboratory use which they make.

Part of this book appeared as a series of articles in *The Chemical Engineer* during 1905. These have been carefully revised and much new matter added.

RICHARD K. MEADE.

May 15, 1908.



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# The Design and Equipment of Small Chemical Laboratories

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## CHAPTER I.

### GENERAL FEATURES.

In designing and equipping a laboratory, two aims must be kept always in mind—First, to promote accuracy, and second, to economize time and labor. Don't make the mistake of sacrificing the former unnecessarily to the latter, but always save time and labor when possible and to that end arrange your laboratory systematically. Nothing so facilitates rapid work as having a place for everything and doing everything in that place. Where space permits, have a separate table for carbon combustions, for ignitions, extractions, evaporations, for titrations, precipitations with  $H_2S$ , distillations and for electro-chemical analysis, filtrations and various and sundry special tests.

**General Arrangement.**—The location of the laboratory is usually controlled by circumstances. Sometimes it is a separate building, sometimes part of an office building and sometimes a room or rooms in the mill or factory set apart for the purpose. If a separate building is made for the laboratory special features can be incorporated in its design. If, however, the laboratory is to be located in an office or factory building, the chemist has usually to content himself with what he can get. The laboratory if possible should consist of three rooms, two of which should be neatly finished inside and intended for the analytical work, and the third of which may be merely a shed room to be used for the preparation of the samples, etc. Where only one room is available for the laboratory, a small room should be wainscotted off a corner of the large one for a balance room. The size of this balance room will usually be controlled by that of the large one. If permissible, it should be large enough for a desk, book-case and balance table. When possible it should always include a window; if not, the upper half of the sides may be made of glass. Indeed, even when a window is present, it is convenient to do this, running the glass low enough to permit of any one

seated at the balance or desk seeing out into the room. In this way the chemist can keep track of things while weighing or writing.

**Location.**—If a choice is offered in locating the building or selecting a room, get as far away from the noise, dirt and vibration of the mill, furnace or smelter as circumstances will permit. If the laboratory is to be located in an office building of two or more stories, there are several things to be said in favor of locating on the lower floor. The chief advantage is having a firm foundation for the balance, such as can be obtained by erecting a short brick or concrete pier. Its disadvantages are usually annoyance to the chemist from business callers for the office, and annoyance to the office force from the fumes rising from the laboratory. However, properly posted signs (using foreign words where foreign labor is employed) in the hallways, and good ventilation and hoods in the laboratory will usually do away with these annoyances.

In the new office building of the Dexter Portland Cement Co., the laboratory is located on the second floor, the balance room being directly over a heavy concrete fire proof vault for the storage of papers, etc. The balances are mounted on concrete piers resting on the vault sides and roof and are free from vibration. This arrangement is excellent and is one which can be followed in many places. A little further on in the book a method will be described for mounting balances in factory buildings, etc., where they are subjected to jar and vibration.

When large samples have to be reduced either by hand or mechanical means this may be conveniently done in the basement of the office building.

**Light and Ventilation.**—The laboratory should be provided with a high ceiling. It should be well lit either from windows or a skylight. The windows should come near the ceiling, so that ventilation can be secured when necessary by lowering the upper sash. If a skylight is used it should be provided with ventilators. In some laboratories artificial ventilation must be resorted to, in which case a fan (run by an electric motor) located in the upper part of one of the windows will help matters.

**Floor, Walls and Ceiling.**—The floor of the laboratory should be of some material, hard wood is best, which can be cleaned easily. Concrete and tile floors are very trying, for the analytical

chemist is on his feet so much that he is apt to find such a hard floor very fatiguing. Concrete floors may be covered with felt or corrugated rubber matting which can be easily cleaned and lessens the fatigue of those standing on it. Old floors full of cracks can be covered with oilcloth or linoleum. This latter makes an excellent floor covering, as it can be easily cleaned.

The walls and ceilings should be finished in some material such as hard wall plaster or cement, which is not readily attacked by acid fumes. If the wall is old and there is danger of grains of sand and lime dropping in beakers, dishes, etc., it is best to cover the plaster with wall paper. This should be of a light pattern, so as not to darken the room. It is probable that wood is really the best covering for the walls and ceilings of the laboratory, as it is not attacked by acid fumes. Metal ceilings are most objectionable, and all exposed metal beams should be well protected by either aluminum paint or asphalt varnish, to prevent corrosion from acid fumes. The electric wiring should always be concealed under the ceiling, as the acid fumes rapidly attack the copper wire, and the lamp socket should be of porcelain.

**Heating Appliances.**—For heating the laboratory, hot water or steam is to be preferred. Air is objectionable from the dust it usually carries. If the laboratory is located near the boilers, steam for heating can usually be obtained from these. The heating apparatus of the balance room should not be too near the balances.

**Interior Arrangement.**—The interior arrangement of the laboratory will depend somewhat upon the kind of work that is to be done. In every laboratory, however, there should be a hood, a sink, a burette table and a bench for general work. In all the laboratories which the writer has designed he has set aside one special stone topped table on which to place the blast lamps and burners used for igniting precipitates, and one special bench to use for long stemmed funnels.

The inside arrangement of a laboratory can best be illustrated by giving plans of several laboratories.

**Laboratory for Iron and Steel Work.**—Figure 1 shows the arrangement of a model small laboratory for iron and steel work, consisting of a one-story frame building with three rooms. The main laboratory is  $20 \times 19$  feet, the balance room  $10 \times 9$ , and the sample room  $10 \times 11$ . The balance room is located at the north-

east corner, giving a north light on the balances. It is large enough to accommodate a bookcase, roller top desk, balance table, and the necessary chairs. The room for the preparation of samples contains a small Bosworth crusher for reducing ores, a drill for making borings, mortar, pestle, etc. The center of the analytical laboratory is occupied by the hood. The details of this are shown in Fig. 4. It is divided into two compartments, so that silica may be determined in one side by Drown's method, while sulphur is being determined on the other by oxidation, without danger of contaminating the latter by the former. The sink is at one end of the double hood and a stone top ignition table at the

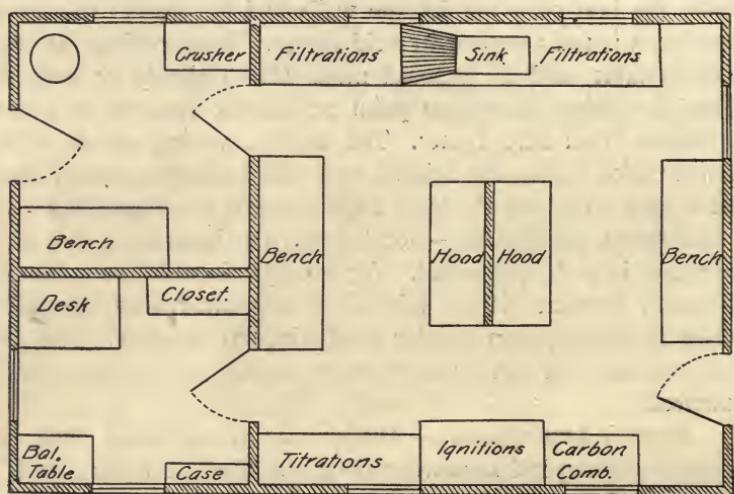


Fig. 1.—Plan of Small Laboratory for Iron and Steel Analysis.

other. Two general work benches face the two hoods, and the ignition table is flanked on one side by the burette table and on the other by the table for carbon combustions, also stone topped. The sink has a table provided with vacuum on each side of it. The advantage of the arrangement with the hood in the center is that the one laboratory is practically divided into two, and two men can easily work here without interfering with each other in any way; each hood is accessible to the sink, to a large work bench, and to the table with the vacuum pumps.

Fig. 2 shows another arrangement of a laboratory  $16 \times 18$  feet. Here a balance room has been cut off from the main room

by a wainscotted partition. It is  $7 \times 8$  feet and does not much more than accommodate the desk and balance table and chairs

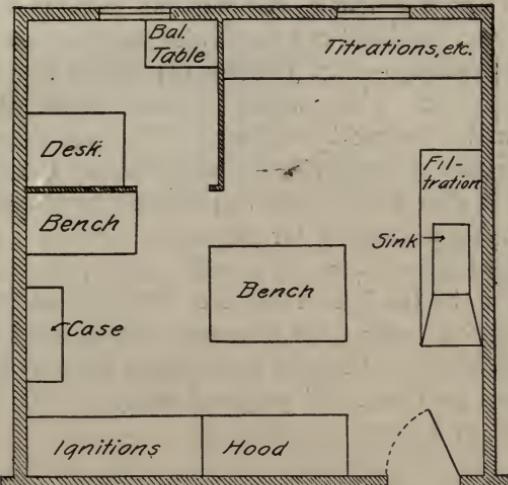


Fig. 2.—Plan of Small Laboratory with Balance Room Partitioned Off.

for the two. It is taken from a corner of the room which gives a north light, and as it takes one of the only two windows of the

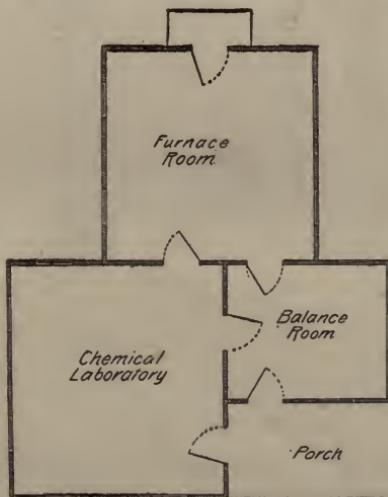


Fig. 3.—Plan of Small Assay Laboratory.

room, the partition is of glass, to help light the main laboratory. The arrangement is evident from the plan. The hood, sink and

large bench are near together, and most of the work is done here, leaving the other benches for special determinations. As the large central bench is double, one man can work at each side of it. If necessary, the hood can be divided into two compartments.

**Assay Laboratory.**—Fig. 3 shows the design of a laboratory for fire assay work. This consists of three rooms, an office, an assay room and a chemical laboratory. The assay room contains a crucible furnace, a muffle furnace and a roaster if sulphide ores are assayed. It should also contain a coal bin, the necessary crushing and grinding machinery for preparing samples, a rough balance for weighing the fluxes for crucible assays, etc. In the office will, of course, be the button balance, the pulp balance and the analytical balance, besides the necessary chairs, desk, cases, etc. The chemical laboratory should be provided with hood, sink and work benches, as usual. If properly equipped this laboratory will take care of a great deal of work.

## CHAPTER II.

### HOODS.

Upon nothing does the comfort of the inmates of a laboratory so much depend as upon the hood, and it is essential that this should "draw" well in order to carry off the fumes.

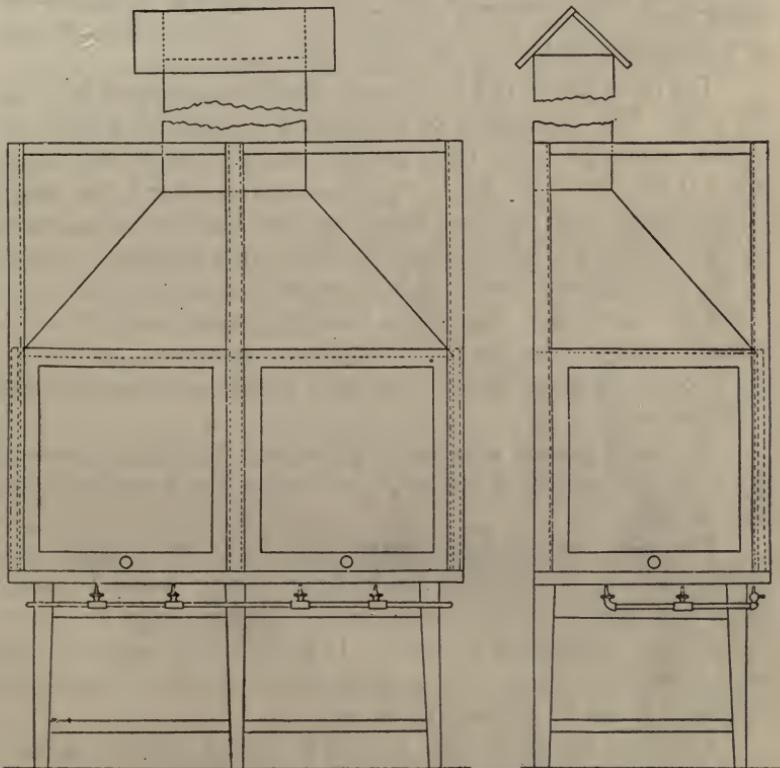


Fig. 4.—Simple Laboratory Hood.  
Scale  $\frac{3}{8}'' = 1'$

**Simple Hood.**—Fig. 4 shows the simplest form of hood and one which will draw best of all, because the hot gases go straight up without making any turns or bends. A hood opening into a flue warmed by chimney gases, even though the hood gases make a double bend, will usually draw. Indeed, for most work it is

unnecessary to encase the sides of a hood built along these lines with sliding sashes. The writer designed some years ago for his use a hood which consisted merely of a stone topped table over which a sloping canopy top was constructed, like a hood to a blacksmith's forge, with a straight flue leading up through the roof. This hood was not connected with the table in any way, even the corner posts being omitted. It stood against the wall. The table top measured 8 x 3 feet and the rim of the canopy stood 6 feet 2 inches from the floor, and projected 3 inches on all sides beyond the table top. This hood drew beautifully, and even sulphide precipitations could be made in it without even an odor of hydrogen sulphide escaping into the room.

The hood shown in Fig. 4 can be readily understood from the drawing. The dimensions of the hood are 6 feet by 3 feet. Its bottom stands 32 inches from the floor and the rim of the canopy top is 6 feet above the floor. In proportioning a hood, the height of the bottom from the floor should be such that the hot plates to be used are on a level with the top of the work benches. The rim of the hood canopy should be high enough from the floor not to touch the operator's head when he stands erect under it. It is usually cheaper, also, to take stock-size sash-frames and design the hood to fit these rather than have special sash-frames made to fit the hood.

The hood shown is meant to go against the wall. If a square hood for the middle of the room is desired, the front and sides will be alike.

The sides of the hood shown are closed by sliding glass frames, which are mounted on Pullman spring balances, doing away with pulley weights, which make a much larger and more cumbersome framework necessary. In this hood the upper framework is made to merely rest upon the table top if this is of stone; otherwise it can be fastened to the top.

The hood shown in Fig. 4 is made with a table for a base. Instead of this, the hood can of course be mounted on a press or cupboard using the latter for storage of acids, apparatus, etc.

**Concrete Top.**—The writer having had considerable experience with concrete, the idea of covering the floor of the hood with concrete instead of using a stone slab occurred to him some years ago. Where this is done, it is only necessary to put a wood top of rough plank on the table, making this top three inches nearer

to the floor than the completed top is to be. Then put on the super-structure and canopy part of the hood. Next finish off the rough edges of the table top and fasten tightly to this with thin screws a wooden rail, whose top is 3 inches from the rough wooden top. Then fill in to the top of this wooden rail with concrete. The best mixture for this purpose is limestone screenings, using one part of cement to 3 of limestone screenings passing through a  $\frac{1}{8}$ -inch screen. The advantage of using limestone screenings in place of sand is that any acids spilled in the hood will attack both the cement particles and those of the limestone, leaving an even surface; while if sand is used, only the cement is attacked, leaving a rough, uneven surface. The cement sand mixture is also more porous than the cement limestone. After the concrete is hard the rail can be removed, the screws greatly facilitating this. Another very serviceable floor for a hood is sheet lead. This can be cut to fit the wood bottom, allowing 3 or 4 inches for turning over and under the sides. One-sixteenth inch lead is thick enough and can be easily worked. The hood may also be lined with tiles or with  $\frac{1}{4}$ -inch asbestos board. The concrete mixture, however, is much cheaper than the latter and is far preferable to it.

**Gas Taps.**—The gas taps for the hood should run around the front of the hood, as it is very inconvenient to reach over hot plates, etc., to turn them out; and at times it may be necessary to control the gas supply from outside the glass doors. The gas taps are usually placed as they are in Fig. 4, the rubber tubing passing to the interior of the hood through holes bored in the hood floor. If this latter is to be of concrete, before putting in this, cut 4 inch pieces of iron or brass pipe  $\frac{3}{4}$  inch inside diameter and set in holes bored through the rough wood bottom so that their tops are level with that of the wooden rail. Another plan is to pass the tubing over the edge of the hood floor, in which event notches must be cut out of the sashes; or, since some air must always be admitted to the hood to form a current to sweep the fumes up the flue, a block or stop may be put in the frame for the sashes so that when closed the bottom of the sash is one or two inches from the stone slab. This is usually only done when a stone top which has not been bored is used as a bottom. Where the top is of concrete the gas piping may be first fitted and laid on the wooden top, the concrete then being placed over it, leaving only the tops sticking up, as in Fig. 5.

**Canopy.**—In the hoods shown, the canopy part is built of  $\frac{1}{2}$  inch matched and grooved lumber. This is light and is not attacked by fumes. It is so far away from the burners as to be in no danger of catching fire. Where the room is not very well lighted this top may of course be made of glass; in which case, it is perhaps best to run the framework up to the ceiling and merely add a row of sashes, making a sort of glass box. The plastered ceiling should be covered with wood, however, as it is almost sure to be attacked by the fumes, dropping sand and lime into solutions, etc., evaporating below.

**Lighting the Hood.**—If the laboratory is lit with electric lights, a few of these should be put in the hood. They should be held in a round porcelain base socket, without key, screwed to the frame, the wiring all being concealed and the lights turned on

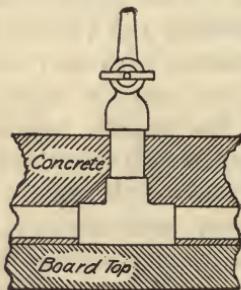


Fig. 5.—Arrangement of Gas Taps in Concrete.

and off by a switch on the outside. The metal work where the globe and socket join should be wrapped with insulating tape so that no metal is exposed. If gas is used, an ordinary fish tail burner in the centre of the hood, directly underneath the wood flue, will not only give the necessary light but will also help along the draft of the hood.

**Mechanical Ventilation of Hoods.**—It occasionally happens that gases given off in the hood are heavier than air and will not rise. In this event, the gases must be drawn off at a level with the hood floor, by a small fan directly connected with a motor. Such a hood should be provided with a small sliding ventilator at the top, which may be opened, admitting fresh air into the hood when the fan is running. It is hardly necessary to say that such a hood should have sides.

Where a hood connects with a flue in the walls of the build-

ing it is sometimes hard to get a draft, particularly in summer time when the flue is cold. In such cases a lighted gas burner placed just inside the flue will help matters. The flue should also draw only from the hood. Draft can of course always be secured by fans; these are rapidly corroded by acid, however, and are expensive to operate. Where their use is necessary and fumes corroding metal are present, the writer suggests washing the gases by passing them up through a cylinder formed by a length

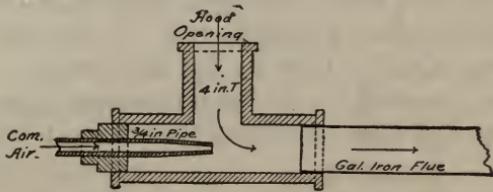


Fig. 6.—Injector for Ventilating Hoods.

or two of sewer pipe. This is to be filled with coke, kept wet by water trickling over it, in order to wash out the corrosive gases, before they come in contact with the fan. The acid water could then be caught in a lead or earthen-ware receptacle at the bottom of the pipe and allowed to go to waste by drawing off occasionally. When high pressure air is available, a small hood may be ventilated by allowing this to flow through a small opening creating suction, see Fig. 6, on the principle of an injector.

## CHAPTER III.

### SINKS AND WATER SUPPLY.

In most college laboratories and in some large industrial ones the sinks are located in the middle or at the ends of the work benches. In a small laboratory, however, it will be found more convenient to have a separate table for the sink and it will save plumbing to fit this table up for filtration by vacuum, distillations, etc.; in short for all the operations of the laboratory requiring water. Figure 7 shows such a table. It is 12 feet long

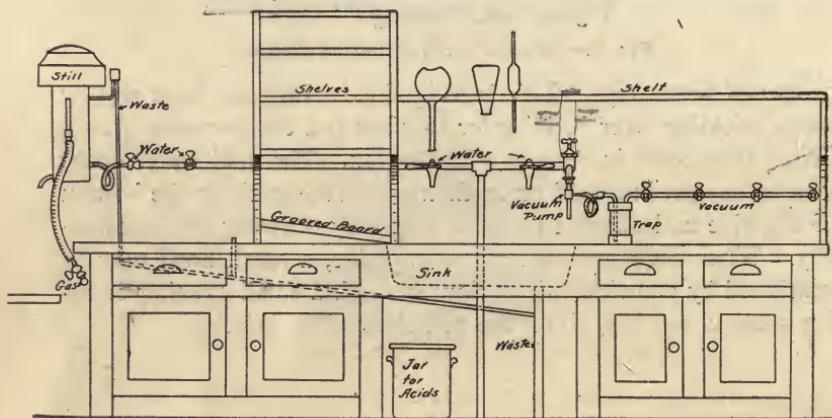


Fig. 7.—Sink and Table.

and 3 feet wide. Its top is 33 inches from the floor. It is provided with a vacuum pump, a sink  $3 \times 2$  feet, two water taps, a drain board, shelves for clean beakers and one for drying flasks, etc., water taps and waste pipe for condensers and for the still for furnishing distilled water, and gas taps for these latter.

**Sink.**—The sink is placed four feet from one end of the table, leaving a space of table of this length on the right, for filtrations by vacuum; and on the left for drying beakers and for standing apparatus to be washed, etc.

The sink itself may be of galvanized iron, zinc or enameled iron. The latter is to be preferred. Wood also makes a very good sink, but it is hard to make one out of it which does not

leak. The writer at one time had a large wooden sink, 4 ft.  $\times$  3 ft., in his laboratory which gave excellent service. In order to prevent drying and consequent leaking, the waste pipe was so arranged as to keep about an inch of water in the bottom of the sink all the time. The usual objection to metal sinks is the corrosion due to acids, and the fact that glassware must be carefully handled around them, as a very light tap against the bottom of a metal sink is sure to result in a broken beaker or flask. A sheet of corrugated rubber matting or a small rubber door mat laid in the sink will save much apparatus. Underneath the sink is as good a place as any for a large stoneware jar in which to pour waste acids and filter papers. This jar is to be emptied daily by the janitor and, if used, will save many plumbers' bills for renewing the waste-pipe.

As the sink table is 3 feet wide and the sink is but two, the latter should be so placed in the former as to be about four inches from the front of the table. This space, before and behind, should be boxed up and the edges of the boards, lapping over the sink, nicely rounded. In the drawing, the waste-pipe is shown straight without the usual S-shaped trap. When the waste-pipe does not lead into the same sewer as the toilet room, this trap can be omitted; and, if it is done, will save repairs, as acids are almost sure to find their way into the sink and, if the acid water lies in the bend of the trap, it will in time eat a hole in this. Lead is to be preferred to iron for the waste-pipe as the less readily attacked by acids.

**Water Taps.**—In the illustration of the sink table a peculiar form of water tap is shown over the sink. This is one made by Thos. Saville, Philadelphia, Pa. An ordinary lever tap may also be used. The advantage of this form is that the water can be turned on full with a single motion of the hand or shut off equally easily. It will be found of advantage to place a few inches of rubber tubing on the end of at least one of the taps. This not only permits the stream of water being directed in any direction but if the force of its flow from the tap is considerable it deadens this somewhat. The taps should stand at least six inches forward of the rear end of the sink and their tips should be about 18 inches from the bottom.

**Drying Apparatus.**—A board for drying beakers, etc., should be located to one side of sink. In the table shown, this board is

2 ft.  $\times$  3 ft., extending all the way across the table. It is corrugated like a wash board, all the grooves radiating from the sink so as to drain the water into this. Above the board are shelves for clean dry beakers. If thought desirable, these can be made into a cupboard by adding doors. Above the sink is a shelf bored with holes of various sizes for drying flasks, pipettes, etc., as shown in the cut.

Casseroles are much used in some iron and steel laboratories in place of beakers. For drying these, a board with pegs, inclined about  $15^{\circ}$  from the vertical, placed behind and so as to drain into the sink, will be found useful. The pegs should be in pairs, in a row, and placed so that the casserole, laying face to the board, handle downward, has a peg on each side of the handle. The pegs should be about four inches long and the rows should be no nearer together than the diameter of the greatest casserole in use. Each pair of pegs should be the same distance apart as the rows, and the individual pegs of the pair should be about  $1\frac{1}{2}$  or 2 inches apart.

**Filtration by Vacuum.**—The vacuum or suction arrangement shown has four openings and hence four filtrations can be made at the same time. If less than this number are ample for the work to be done, the table may be shortened accordingly. In an iron and steel laboratory, where many samples of pig iron are analyzed for silicon, sulphur, etc., four will not be found too many, as one of them may be needed for a reductor and the rest will be kept in use for filtering off graphite, etc., from the solution of the pig iron. Since three or four filtrations can usually be carried out as rapidly as one, it will save time to provide means for making this number simultaneously.

**Vacuum Pump.**—The form of vacuum pump, filter pump or aspirator shown in the illustration is that of Chapman. This pump is small but effective. Figure 8 shows the details of its construction. It is made in three sizes. A No. 2 pump will be of ample capacity to take care of four filtrations and the smallest size, No. 1, will furnish vacuum for one or two filtrations. It can be obtained with a threaded coupling to fit a threaded faucet or with a smooth coupling for a plain faucet. The former is much to be preferred and is essential if the water pressure to be used is great. The threaded coupling has a milled ring around it so the pump can be readily removed from the faucet for cleaning.

This is sometimes necessary, as it often becomes stopped up with sticks, leaves and other trash in the water. The greater water pressure which is at hand the more perfect vacuum can be obtained. A fair suction will be secured, however, from a head of water such as would result from a tank placed on the laboratory roof. Another form of air pump is that of Richards. This also is very efficient and is made in two sizes. Still another form has a vacuum gauge attached, but these cost considerably more. If it is desired to measure the vacuum, it may be done by means of a long U tube, about 30 inches high, half full of mercury. One end of this is attached to the pump and the vacuum is measured by the difference between the levels of the mercury in the two limbs of the tube. If wished this mercury tube may be fastened

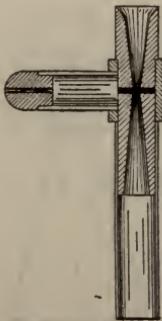


Fig. 8.—Chapman's Vacuum Pump.

to the wall behind the sink table and permanently attached to one of the openings of the vacuum line.

As these little pumps are somewhat apt to let water pass back into the air line, if the water supply is suddenly cut off or the pump clogs, it is safest to place a trap between the pump and the air line. This may be merely a pint bottle provided with a double perforated stopper, through one of whose holes a piece of glass tubing passes nearly to the bottom of the bottle and through the other of whose holes a shorter piece of tubing reaches just below the stopper. A more durable form of trap is made of a piece of pipe 3 inches in diameter and 6 inches long capped at both ends to form a small drum. Two pieces of  $\frac{1}{4}$ -inch pipe are fitted into this, one reaching nearly to the bottom of the drum and the other just inside the cap. The long pipe or tubing connects with the pump and the short one with the air line. This latter

joint if the drum is used should be made by a threaded pipe fitting, and the connection between the drum and the pump by a piece of heavy walled rubber tube, as shown in Fig. 7, to facilitate disconnecting the pump for cleaning.

Where a powerful suction is required and there is only a light head of water, these filter pumps may be operated by steam or compressed air. Schutte & Koerting Co., Philadelphia, Pa., make a very powerful filter pump to be operated by steam which they call the "Universal Steam Jet Laboratory Exhauster." This with vacuum gauge attached is listed by the manufacturers at \$10.00. It requires for operation a volume of steam equivalent to an evaporation of 12 pounds of water per hour. When there is a head of water available, however, the writer knows of nothing

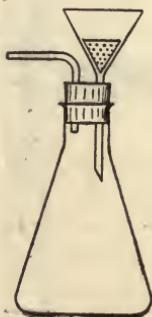


Fig. 9.—Flask Equipped with Funnel and Cone for Filtration by Suction.

better for filtrations by suction than the Chapman filter pump first mentioned.

**Method of Using Vacuum for Filtering.**—For use with these pumps in filtering nothing is more serviceable than a thick walled Erlenmeyer flask fitted with a two-hole rubber stopper. A 60° short, thin-stemmed funnel, of exact angle, passes through one hole of the stopper and a piece of glass tubing bent at right angles through the other, reaching just below the stopper. A perforated platinum cone fits in the funnel and prevents the filter paper from being torn by the suction. Figure 9 shows the completed apparatus. A special form of Erlenmeyer flask can be obtained, having a tube inserted in the neck and made of very heavy glass, at a slightly increased cost. This is, however, but little more convenient and indeed has some disadvantages over the simpler form. In using Erlenmeyer flasks, the filtrate has often to be transferred

to a beaker. In order to obviate this the bell-jar and ground glass plate shown in Fig. 10 can be used. For filtering barium sulphate the Gooch crucible is very useful. This requires suction and consists of a flat bottomed perforated crucible provided with a cap, see Fig. 11. The perforated crucible is placed in one end of a piece of soft rubber tubing of a large bore, the other end of which is stretched over a small funnel passing into the Erlenmeyer flask, through a rubber stopper, see Fig. 12. The method of using is as follows: Pour a little prepared asbestos (purified by washing with hot concentrated hydrochloric acid) suspended in water into the crucible and attach the suction to

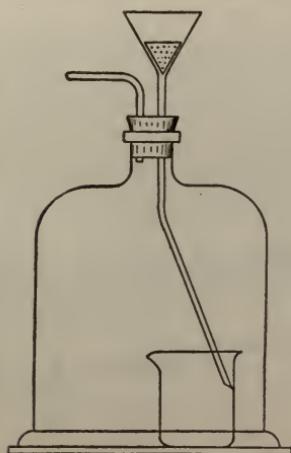


Fig. 10.—Bell-Jar and Plate for Filtration by Suction.



Fig. 11.—Gooch Crucible.

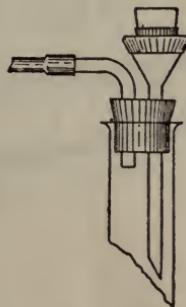


Fig. 12.—Gooch Crucible Ready for Use.

the flask. The asbestos at once forms a thick felt over the bottom of the crucible, which by using the suction may be readily washed with water. After washing, suck as dry as possible with the pump. Remove from the funnel, detach any pieces of asbestos that may be on the outside of the bottom of the crucible, cap, ignite and weigh. Remove the cap, attach to the funnel as before, apply the suction and pour the liquid to be filtered through the crucible, wash cap, dry, if necessary, ignite and weigh as before. The Gooch crucible is made of either porcelain or platinum; those of the former material have no cap, and are made either with a permanent bottom, or with a movable porcelain or platinum plate, which rests on a small border, in place of a bottom.

When the filtrates are to be discarded and many filtrations are made every day, such as silicon in pig iron, it will be found more convenient to filter into a large bottle, instead of a small flask, doing away with the necessity of emptying the receiver after each filtration.

**Stills, Extractors, Etc.**—The left end of the table shown in the cut is reserved for distillations and is accordingly provided with gas as well as water. Where many distillations or extractions have to be made, however, a larger table may be found necessary. Figure 13 shows a compact still for nitrogen determinations, which will allow of six distillations being carried on at once, and which will fit this table space nicely. Stutzer's ap-



Fig. 13.—Apparatus for Nitrogen Determination.

paratus for extractions is also very compact and is used in the German agricultural experiment station at Bonn. It will take care of six extractions. Both forms are made by Messrs. E. H. Sargent & Co., of Chicago.

The cooling water from the condensers runs down into a waste pipe, which is brought up flush with the top of the desk, behind the drawers, and which connects with the drain pipe of the sink, as shown in Fig. 7.

Where extractions have to be frequently made with ill smelling liquids, such as carbon bisulphide, it is well to either partition this end of the table off to form a hood or else to extend the water and waste line into the hood. Where extractions are made

with very volatile or inflammable liquids, it is safest to do the heating with electric stoves. Several forms of these will be mentioned later on.

**Miscellaneous.**—The sink table as illustrated is provided with drawers and is boxed up to form a closet for storage of apparatus. To prevent trash, etc., being brushed into the latter when the floor is swept, the floor of the closet should be at least 3 inches above that of the room.

The top of the table may be of wood, stone, slate, concrete or sheet lead. Two-inch white pine board makes a good cheap top. When this becomes acid scarred it can be covered with oilcloth or the boards turned, exposing the lower surface, or renewed entirely. The desk itself should be filled and varnished or primed and painted. The top, however, if of wood should not be treated in any way but should be left just as it is.

It is well to have on the water line, somewhere easily accessible, a valve with about five or ten feet of good garden hose, with nozzle attached, for fire protection. This will be found cheaper than the hand grenades and equally serviceable for putting out small fires.

## CHAPTER IV.

### DESKS.

**General Arrangement.**—The one thing about a mill laboratory which is very similar to the college laboratory is the desk or work-bench for general operations which is present in one form or another in every laboratory. If the desk is to be placed in the middle of the room, it is usually a double desk; if against the sides of the wall, it is, of course, a single desk. The size will



Fig. 14. Laboratory Desk—Front Elevation.

vary to suit the space which it is to occupy. Particularly is this true with respect to length. With regard to its width, the latitude is, of course, not so great. Nothing is gained by having a single desk over three feet wide, or a double desk over six feet wide; nor would it be advisable to make a double desk less than forty-five inches wide. The height from the floor may vary with the ideas of the user; the usual height, however, is from 34 to 38 inches. The writer has found 36 inches a good height. The general work-bench is usually made with closets and drawers. Some desks are made with the drawer part projecting beyond the

closets. This has the advantage of making the closet less deep and consequently allowing several shelves to be put in the latter.

Figures 14 and 15 show a desk such as the writer has generally used, which is modeled somewhat after those with which most students are familiar in the college laboratories, and Fig. 16 shows this same desk with the drawer part projecting beyond the closets. Its dimensions, in this particular case, are 7 feet by 5 feet and it is 36 inches in height. It has five drawers on each side—two narrow drawers for watch glasses, filter papers, evaporating dishes and such flat apparatus; one deep drawer for large,

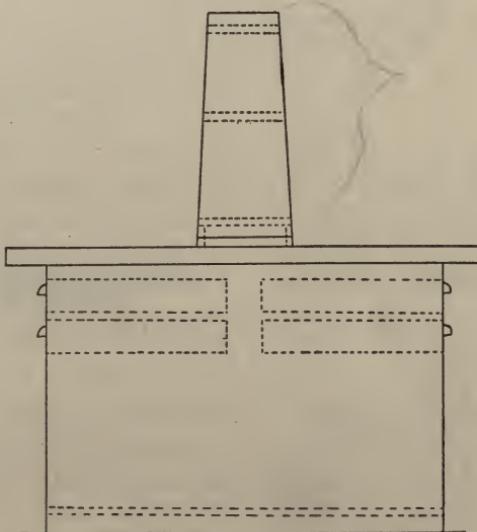


Fig. 15.—Laboratory Desk—End Elevation.

bulky articles; and two wide shallow drawers for burettes, glass tubing, and long flat articles. If the desk is desired longer than this, it can, of course, be lengthened by the addition of another set of drawers and cupboards.

**Drawers.**—There is usually more or less inconvenience in pulling drawers in and out, from their binding on the sides. This is particularly annoying when the drawer is full of glassware, as breakage is likely to occur from the sudden jar used to force it in or pull it out. Figure 17 shows an arrangement which will prevent this. It consists in fastening a narrow strip of well-seasoned board, *B*, along the bottom of the drawer, *A*, and fast-

ening to the frame of the desk two other narrow strips, *C*, *C*," one on each side of the strip on the drawer. These two strips form a groove in which the other one runs and in this way the drawer is always kept in the proper position to slide in and out easily.

The drawers can be cut up into partitions for different sizes of watch glasses, filter papers, evaporating dishes, etc. If no safe is at hand in which to store the platinum, one of the top drawers of the bench may be provided with a lock and key and used for this purpose. In this case, instead of an ordinary bottom it should

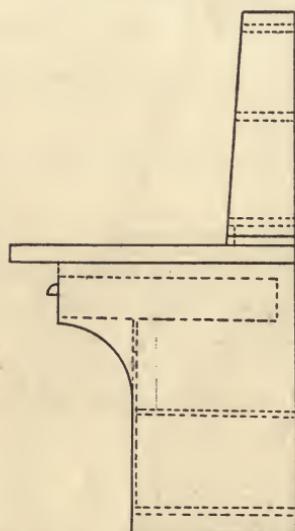


Fig. 16.—Laboratory Desk with Projecting Drawers.

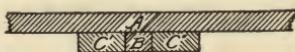


Fig. 17.—Guide for Drawers.

have a piece of two-inch board fitted in. In this are to be bored holes which exactly fit the various crucibles, dishes, etc., in use in the laboratory. The holes for the crucibles need not be gouged out just to fit the crucibles but may be bored with brace and bit of such size that the crucible will fit in them snugly. Alongside of the hole for the crucible should be cut a narrow deep slot for its lid. This is a very satisfactory way of keeping platinum, as each crucible has its place and can not roll around in the drawer, thereby getting out of shape.

In the drawers designed for such apparatus as U-tubes,

$\text{CaCl}_2$  tubes, etc., it will be well to have rows of pegs about three or four inches apart, and  $1\frac{1}{2}$  to 2 inches high; the tubes, etc., can then be laid in among these in such a way as to keep them from rattling around every time the drawer is opened, thereby preventing breakage.

**Cupboards.**—The floor of the cupboard should be three inches above the floor of the laboratory to prevent dust, etc., being swept into the former. Shelves can, of course, be added if desired. In the desk shown in Fig. 15 there is a shelf midway between the floor and the cupboard. In a desk made as deep as this, one shelf is all that can well be added. In a narrower desk, several shelves can be added, as it is not necessary to reach back so far for the apparatus. Instead of having the doors of this cupboard arranged to open outward, as they are in the cut, they can be fixed to slide by each other in grooves.

**Shelves.**—The reagent shelf should be at least six inches wide for the single desk and about ten to fourteen for the double desk. A six-inch shelf will hold a five-pound reagent bottle or a five-pint acid bottle. If wished, the bottom shelf may be six or eight inches wide, and the next an inch or so less. If gas is on the table, the bottom shelf may be raised a little above the desk and the space between the two filled in to make a sort of stop, as shown in Fig. 15, so that Bunsen burners cannot be carelessly pushed under the shelves and set them on fire. In most commercial laboratories where special tables can be provided for each operation, gas may not be needed on the general work table. If desired, however, it can be run either along the front of the table just above the drawers, or along the back of the first reagent shelf; or this shelf may be raised six inches from the table and the gas pipe run under it.

If it is desired to set five-pint acid bottles on the shelf, it must be at least 14 inches from the one above it. Nothing is gained by running the reagent shelves higher than can be reached by the operator standing on the floor. The double desk should have a rail running lengthwise down the middle of each shelf, except possibly the top, to prevent things put on one side from shoving things off on the other.

Fig. 18 shows a convenient way of mounting shelves with iron pipes. The construction is evident from the illustration and all parts are regular stock pipe fitting. It is sometimes convenient to

hang shelves from the ceiling so as to leave the whole surface of the table free. If this is done, the above way of using pipe is convenient. These hanging shelves are usually made quite wide and do not come nearer to the top of the table than  $2\frac{1}{2}$  to 3 feet. It is sometimes found more convenient to bring gas down from the ceiling to the single or double tables—running down one pipe on each side of the reagent shelves and terminating each in a double jet. The table may, of course, be provided with water supply and waste pipes and suction if thought necessary. The

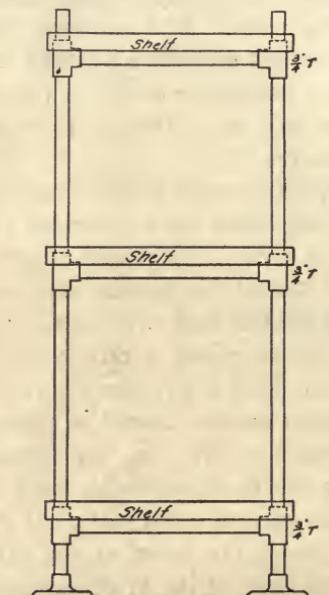


Fig. 18.—Shelves Mounted on Piping.

water may be brought down from the ceiling also, just as the gas is.

**Reagent Bottles.**—A neat form of glass stoppered reagent bottle for this table is that with flat hood shaped stopper. A still better form is one devised by Prof. Jewett, of Oberlin College (see Fig. 19), manufactured for E. H. Sargent & Co., Chicago, Ill. In this bottle, the mouth and lip are completely protected from dust by the hood stopper and its pendant flange, made in one piece. This helps to keep the contents of the bottle pure. These bottles have chemical names and symbols in raised letters with ground

surfaces, made in 4 and 8-oz. sizes. One of the best forms of label which the author has seen is that which Whitall, Tatum & Co., of Philadelphia and New York, have introduced. This is a



Fig. 19.—Jewett's Reagent Bottle.

label of vitrified glass in which the label itself presents a smooth white glass background, against which the transparent letters and symbols show distinctly. This label is more legible than the ordinary raised letter with the ground glass face.

## CHAPTER V.

### TABLE AND APPARATUS FOR RAPID FILTRATION.

**General Features.**—Figs. 20 and 21 show a table especially designed for the use of long stem funnels. It is merely an ordinary table, thirty inches high, provided with a shelf running the entire length across its front, eighteen inches below the top of

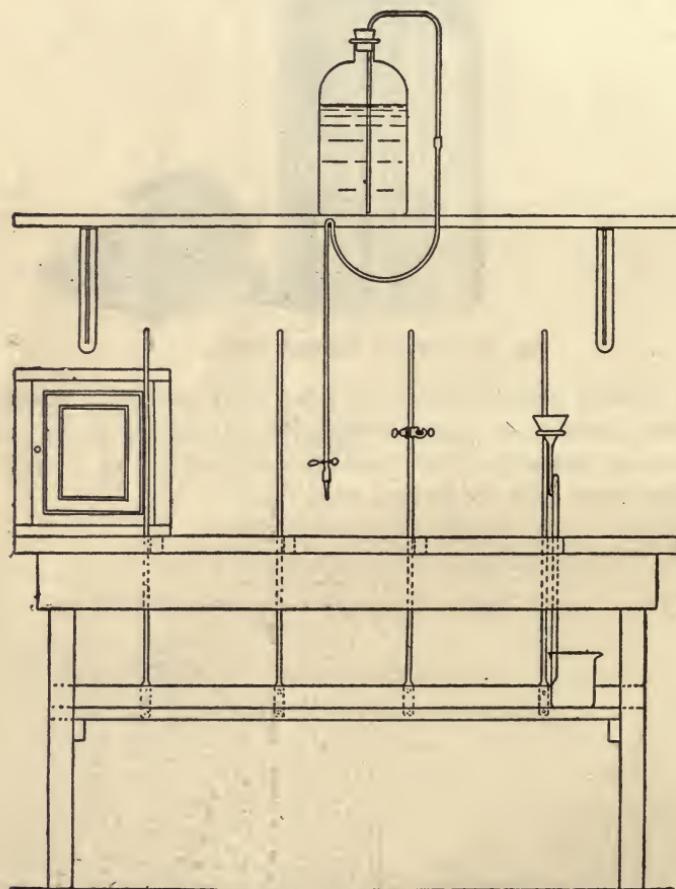


Fig. 20.—Table for Rapid Filtration—Front Elevation.

the table. The shelf is designed to hold the beakers which are to catch the filtrates. The funnels pass through slots, 1" x 2", in the table top and are held in the arrangement shown in the illustration. This consists of an iron rod, screwed fast to the lower shelf of the table and passing up through the top, twelve or sixteen

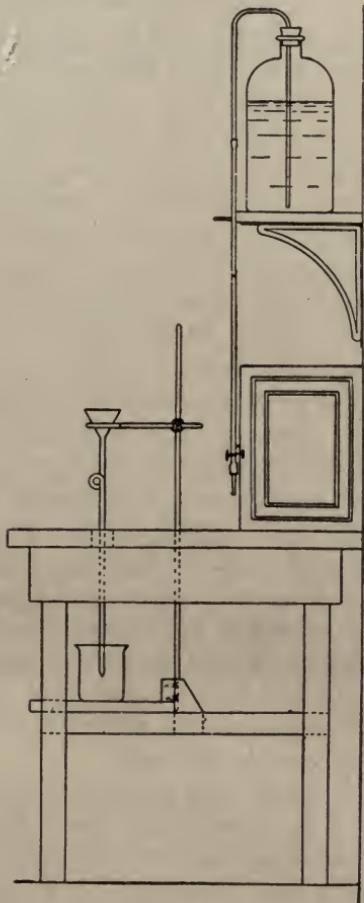


Fig. 21.—Table for Rapid Filtration—End Elevation.

inches or even higher. The funnels themselves are held in glass triangles made of heavy glass rod,  $\frac{1}{4}$ " in diameter, which in turn are clamped to the rods, at any height desired, by means of right-angled clamps. These filter stands are very neat and are cleaner than the wooden ones. A piece of rubber tube should go

around the glass triangle where it is gripped by the clamp. The funnel stems should be bent in a loop so as to keep them always full of liquid. Instead of making the bend, the tube may be merely drawn out slightly, reducing the diameter to about 1-16 inch. In order to have them fill rapidly it is essential that the filter paper be put in tight, so that no air can leak past. In connection with this table, the writer has used to advantage the device for washing precipitates shown in the cut. It consists merely of a bottle standing on a shelf, either fastened to the wall or supported from the table, provided with a siphon tube to which is attached a long flexible rubber tube terminating in a jet and closed by Mohr's pinch-cock. This apparatus will be found very useful for washing such precipitates as magnesium pyrophosphate, phospho-molybdate, etc. The small cupboard at the back of the table is for filter papers. It may be dispensed with entirely and the japanned tin filter cases, sold by dealers in chemical apparatus, substituted. These latter hold five sizes of filter papers, ranging from 19 cm. down.

**Wash Bottles.**—For covering the necks of wash bottles to be used for hot water, sheet cork will be found the neatest material. It is a good non-conductor of heat and does not flake off on the hands as does asbestos. It is usually sold in plates  $8 \times 4$  inches and  $\frac{1}{8}$  inch thick. A piece of this is cut so as to cover the neck of the flask with about an inch lap. One end of this piece is then shaved with a sharp knife until it tapers to a thin edge, and the cork is placed in hot water to soak. It is then partly dried on a towel, and curled around the neck of the flask. Mucilage or glue is used to hold the two ends of the cork together, string being wrapped around until this dries, which can be hastened by boiling water in the flask.

Asbestos paper is much used for this purpose also. The author's objection to it has always been that it flakes off on the hand and these flakes are liable to get into the filter paper or solution. Where asbestos is used, therefore, it is safest to cover this with cloth. The asbestos is wet and wrapped around the neck of the flask and partially dried. A strip of flannel or felt is then pasted around this so as to entirely cover the asbestos.

## CHAPTER VI.

### IGNITION TABLE AND APPARATUS FOR IGNITIONS.

**General Features.**—In the scheme which we outlined in the opening paragraph of this book, of having a place for everything, it was stated that there should be a separate table for ignitions. The size of this table will, of course, depend on the number of ignitions which are to be made at one time. Ten or twelve can easily be carried out at the same time on a table with a top  $3 \times 4$  feet. There is nothing particularly different about the ignition table from any other table in the laboratory. It should, of course, have a concrete, stone, slate or lead top and can be made similar to the hood table described in Chapter II. Nothing is gained by having a very wide table. If in the middle of the room, a table three feet wide is of ample width, while two feet will do for one set against the wall. The gas tubes should be frequent and there should be some mechanical means for supplying air to the blast lamps. Double gas jets every foot will allow an ignition every six inches. The gas pipes should be run around in front just below the top, as in the hood table.

**Crucible Supports.**—Tripods may be used for holding the crucibles; or ring supports, sold by all dealers, can be used. These latter are to be preferred, as the height of the crucible above the flame can be regulated. They cost, however, about four or five times as much as the tripods. In getting supports, those with triangular bases take up a little less room than the other kind. If a concrete top is to be used, the cheapest and best way of supporting the crucibles is by iron rods projecting from the concrete top, from six to seven inches apart and ten to twelve inches from the edge of the table. Each rod should project fourteen inches above the table top. To do this, cut the rods of the proper height and insert in an upright position in the rough wood top of the table before the concrete is poured on. This latter will of course run around them and hold them very firmly in position. The crucibles are then held at any height desired by means of ring clamps such as are used with supports.

The best triangle to use with the crucible is one of platinum. Its first cost is of course high, but they will stand, if carefully used, any amount of wear. The platinum triangle for a 15 CC. crucible weighs about 8 grams and one for a 30 CC. crucible will weigh about 12 grams. Special clamp supports for platinum triangles are made. These hold the triangle firmly in place and prevent the wire from bending down. They also require much less platinum wire than the ordinary form, three to five grams being sufficient for ordinary crucibles. Nothing is gained by buying platinum wire and bending triangles from this. The best

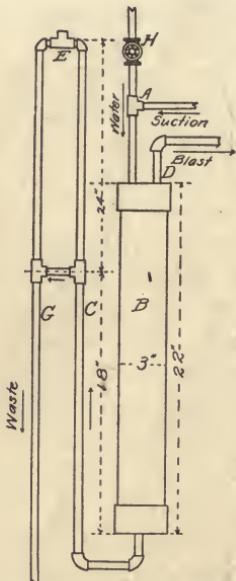


Fig. 22.—Water Blower  
for Blast Lamp.

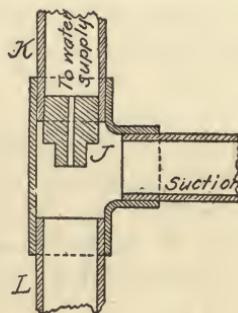


Fig. 23.—Details of the Aspirator  
for Water Blower.

of the pipe stem triangles are those made of iron covered with flanged pipe stems.

**Blast Lamp.**—Every laboratory should have a blast lamp, since its use saves much time and simplifies many operations of the laboratory, and indeed certain determinations (such as silica) cannot be performed correctly without one. They can be obtained in many forms. Bunsen's is one of the oldest and best of these, and it is illustrated in all catalogues of chemical apparatus. It is so fixed that the direction of the flame can be altered by turning a thumbscrew. Another good blast lamp is Weis-

negg's, which is mounted on ball joints. It can be turned about a greater range than Bunsen's, but is also more expensive. The upright blasts cost about as much as the adjustable ones and are not so useful. It is generally better to play the flame at an angle upon the bottom of the crucible than directly upon it, for in the former method the products of combustion are swept away from the crucible, while in the latter they are carried up and around it, entirely surrounding the crucible and cutting off the supply of oxygen from the latter, possibly causing a reduction, etc., of the precipitate being ignited. For this reason the adjustable blast lamps are better than the upright ones. Crucibles to be ignited over the blast lamps should be placed with their bottoms projecting through a round hole in a piece of platinum foil, which in turn rests upon a piece of asbestos board with a slightly larger hole cut into it. While this method of supporting a crucible is not essential and indeed is not the one generally used, it will probably give better results in all cases where a precipitate is to be ignited to a constant weight or when reduction by or absorption of the products of combustion by the precipitate is likely to occur.

**Air Blast.**—For supplying air to blast lamps, every chemist is familiar with the foot bellows. No manufacturing concern, however, can afford to use their chemist's time for pumping a foot blower, so it is more economical to arrange to have some mechanical means of doing this work. If high pressure air is used around the plant, it may be run into the laboratory for this purpose. In blast furnaces, the air from the blowing engine; in machine shops, air for the supply of pneumatic tools, etc., is usually at hand. If the pressure is high, reducing valves should be put in between the laboratory and the compressor or blowing engine.

The writer has in his laboratory a blower which gives excellent service and is working under a water pressure of about 60 lbs. While there is nothing new about it, still it may be worth describing here.

Its construction is shown in Fig. 22. Referring to this illustration, *A* is the aspirator whose detail construction is shown in Fig. 23. *J*, Fig. 23, is an ordinary half-inch T. *K* is a piece of half-inch pipe connected with the valve *H*. Into the tube, *K*, is screwed a small tap through the middle of which has been

bored a 1-16 inch hole. *L* is another piece of half-inch pipe leading to the drum, *B*. The third opening of the T can be connected with the line for aspirating as shown. The drum *B*, Fig. 22, consists of a three-inch pipe, 22 inches long and capped at both ends. *C*, *G*, and *E* are all made of half-inch pipe. *G* leads to the waste. The air for the blast lamps is drawn off at *D*. This apparatus will easily give sufficient air for two blast lamps. It can be purchased, if desired, in a little neater form, from several dealers in chemists' supplies. A small drum may be placed after the blower to catch any water splashed into the air line. One may be made by capping a piece of 3-inch pipe, 12 or 14 inches long, at both ends. It should stand upright and the air should enter and leave at the upper end, and there should be a small tap at the bottom for drawing off any water that may collect in the pipe. The water blower in use by the writer, however, seems to give air free from water and no trap would be needed with it.

In place of the water blower described, a small automatic air pump such as is used for forcing beer from the cellar into the bar, and by physicians and barbers, may be used. It should be of the piston variety, double acting and automatic. A large receiver or drum should follow this to equalize the pressure. A small Root blower or a Crowell blower run from a shaft or by a motor may be used, but either is somewhat noisy if placed in the laboratory and like all machines needs repair and attention.

Dr. Porter W. Shimer described in the *Chemical Engineer* for April, 1905, an apparatus for producing either blast or suction. It consists of a No. 16, "Goulds Air-Pressure or Vacuum Pump" (manufactured by The Goulds Manufacturing Co., Seneca Falls, New York). It is a hand pump, operated by a wooden lever about five feet in length, and, by proper arrangement of valves, it can be used either to compress air or to create vacuum. The diameter of cylinder is 6 inches and the stroke is 10 inches. The displacement of free air per stroke is 280 cubic inches. The inlet and outlet are 1 $\frac{1}{4}$ -inch pipes.

The pump is connected with two boilers such as are used for hot water in connection with kitchen ranges. These boilers need not be new, for rejected ones may be made, with a little repairing, as good as new ones for this purpose. The cylinder for

blast in Dr. Shimer's laboratory is 6 feet high and 18 inches in diameter. The one for suction is 5 feet high and 14 inches in diameter. The manner of making the connections and the position of the valves are shown in Fig. 24. •

By a proper arrangement of the valves the pump may be set for vacuum and the air may be exhausted from the other cylinder for purposes of filtration. By this means any degree of vacuum desired for hastening filtration may be obtained. This cylinder and the piping should be coated inside with acid-proof asphaltum paint to protect it from the corrosive action of acid fumes drawn in during filtration of strong acid solutions. This may also be guarded against by sucking the air through two bottles partially filled with water containing a little caustic soda. The entrance tube to each bottle should pass below the surface of the liquid.

Both blast and suction cylinders may be connected with piping containing as many valved outlets as may be desired.

**Ignitions in a Muffle Furnace.**—Small muffle furnaces, such as are used in assaying gold and silver ores, are often used in large laboratories. When many ignitions are to be made they undoubtedly have some points in their favor. In most small laboratories, however, they are uneconomical, as the gas or gasoline required to heat them up is considerably more than that which would be required to ignite a dozen or so precipitates over Bunsen burners and blast lamps. If heated by coal they are a nuisance and a source of dirt. When fire assays are also made the muffle is of course convenient for ignitions. They are described in Chapter XIII.

**Platinum Crucibles.**—These should be made of the best hammered ware free from alloy. They usually weigh as much, lids included, in grams as they hold in cubic centimeters—that is a 15 c. c. crucible with lid should weigh about 15 grams. For ordinary ignitions a 15 c. c. crucible will be large enough, and, indeed, where 9 cm. filters are used and small precipitates are collected, even 10 c. c. crucibles will answer satisfactorily. For fusions, a 30 c. c. crucible will be found sufficient. For ignitions it is a good plan to have all of the crucibles weigh approximately between 15 and 16 grams, as by this means, considerable time is saved in weighing a lot of them, one after the other, because the large weights on the balance pan do not have to be changed.

The crucibles may be numbered with a small steel die, or, if

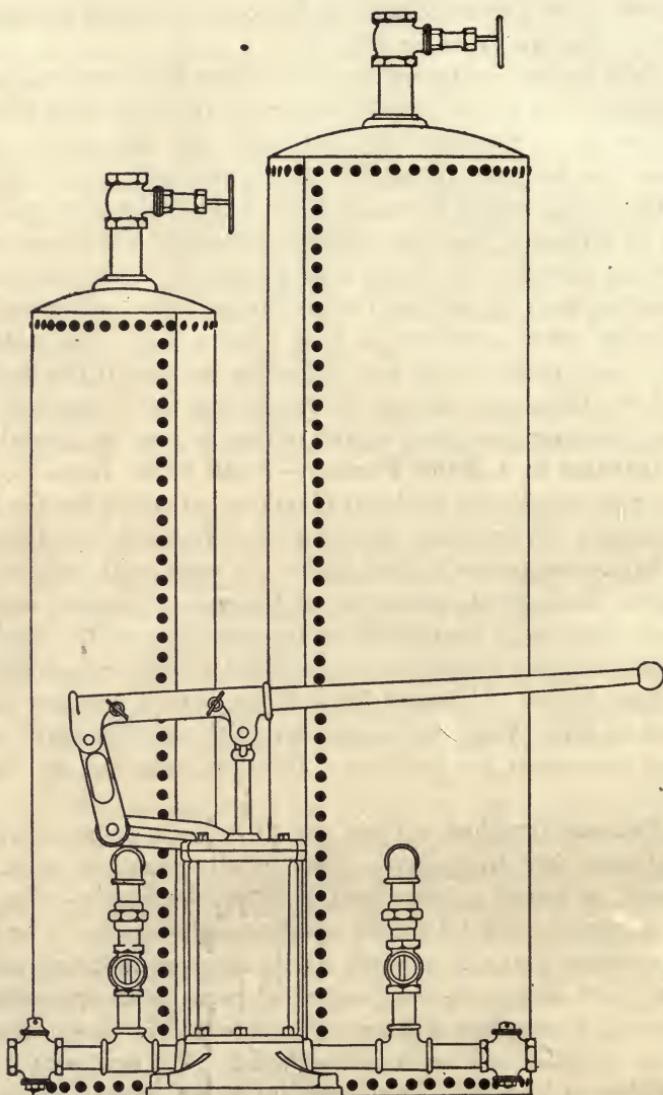


Fig. 24.—Pump and Receivers for Blast or Suction.

there are only a few, this may be done with dots; thus, ::, ::, etc. A wooden mould and plug should be provided and the crucibles kept free from dents and distortion by shaping them up in this. If a mould can not be obtained, a recess casting of the crucible in plaster or cement may easily be made, when it is new, and the end of a broomstick rounded off for a plug to fit inside.

**Porcelain Crucibles.**—Platinum crucibles can not be used for the free metals, the easily reduced metallic oxides and the salts of the heavy metals, such as lead, tin, bismuth, etc., and for igniting such precipitates porcelain crucibles must be resorted to. When platinum can be used, little except first cost is saved by employing porcelain crucibles, as the breakage, etc., of these latter is greater than the wear and tear and interest on the former. Porcelain crucibles may be obtained in sizes ranging from a few cubic centimeters' capacity up to that of several hundred c. c. They are made of both Royal Berlin and Royal Meissen porcelain, the former being the best and costing considerably more than the latter. The Royal Berlin porcelain crucibles are of a more squat form than those of the other makes.

The crucibles may be obtained with vitrified consecutive numbers or letters on the side, by which to distinguish them, at a slightly higher price than the unmarked; or they may be numbered with a lead pencil under the unglazed bottom, before igniting and weighing, the ash of the lead burning into the crucible and making a permanent mark.

Porcelain Gooch crucibles may also be purchased and these are handy for some precipitates. Those with the movable bottom may be used to advantage to collect the carbon for combustion in steel analysis. When precipitates have to be heated in an atmosphere of hydrogen sulphide, hydrogen, etc., Rose's form of crucible may be used. This consists of a porcelain crucible with a perforated cover through the hole of which a porcelain tube passes. When this form of crucible is not at hand an ordinary crucible may be made to serve, being covered by inverting over it an ordinary clay pipe, the gas being led in through the stem of the latter.

**Crucible Tongs.**—These are made in a number of patterns and of a number of metals. For ordinary ignitions, in a platinum crucible, over a burner, the so-called double bent tongs will be found most useful. They allow the removal of the lid from the

crucible and the tilting of the latter on the triangle. For removing crucibles from a muffle furnace, Julian's crucible tongs may be used. Blair also has devised a special form of tongs, scissors shaped, in which the curved and bent part of the tongs is of platinum.

A good pair of tongs is one made of solid nickel or German silver and having platinum shoes on the ends. The shoes are to be preferred to the tips riveted on. The tongs may be obtained single or double bent, the former being the most convenient. In transferring a crucible from the triangle to the desiccator with these tongs, the lid is slipped to one side, just far enough to allow the edge of the crucible to be gripped by the point of the tongs. Where a cheaper pair of tongs is desired, steel forged, nickel-plated tongs are the best.

**Miscellaneous.**—Desiccators, in which to cool crucibles before weighing, are of a number of forms, Scheibler's is perhaps the best. This may be obtained in a number of sizes, of which the 6-inch is probably most suited to laboratory purposes, as it can be carried back and forth from the ignition table to the balance room. For holding the crucibles in the desiccators, porcelain or aluminum plates can be purchased. The latter cool the crucible quicker as the metal carries off the heat faster. Large desiccators may be had and, when these are to be left in the balance room, are found useful. The porcelain plates which come with large desiccators are usually perforated with holes entirely too small for crucibles. If the dealer is furnished a rough drawing, showing size and number of holes desired, he will usually make an aluminum plate at small additional cost. Calcium chloride is used as the moisture absorber in all portable desiccators.

It is often more convenient to lay precipitates aside as they are filtered and ignite later on; an 8-inch bell glass of the low form resting on a glass plate will be found convenient for keeping these out of the dust. The paper may be marked, either before being placed in the funnel or when taken out, with a soft lead pencil. If an acid dish or basin containing a little strong sulphuric acid is placed under the bell jar, and the precipitates are placed in a watch glass resting on the acid dish, the acid will dry the precipitates and filter papers, provided they are left in long enough.

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## CHAPTER VII.

### TABLE AND APPARATUS FOR TITRATIONS.

**Burette Stand.**—A neat burette table is shown in Fig. 25. It is 32 inches high, 4 feet long, and  $2\frac{1}{2}$  feet wide. It has a shallow cupboard below and a narrow drawer. The bottom of the drawer extends only half way across the table, so that it does not interfere with the tubes leading from the burettes to the bottles. The

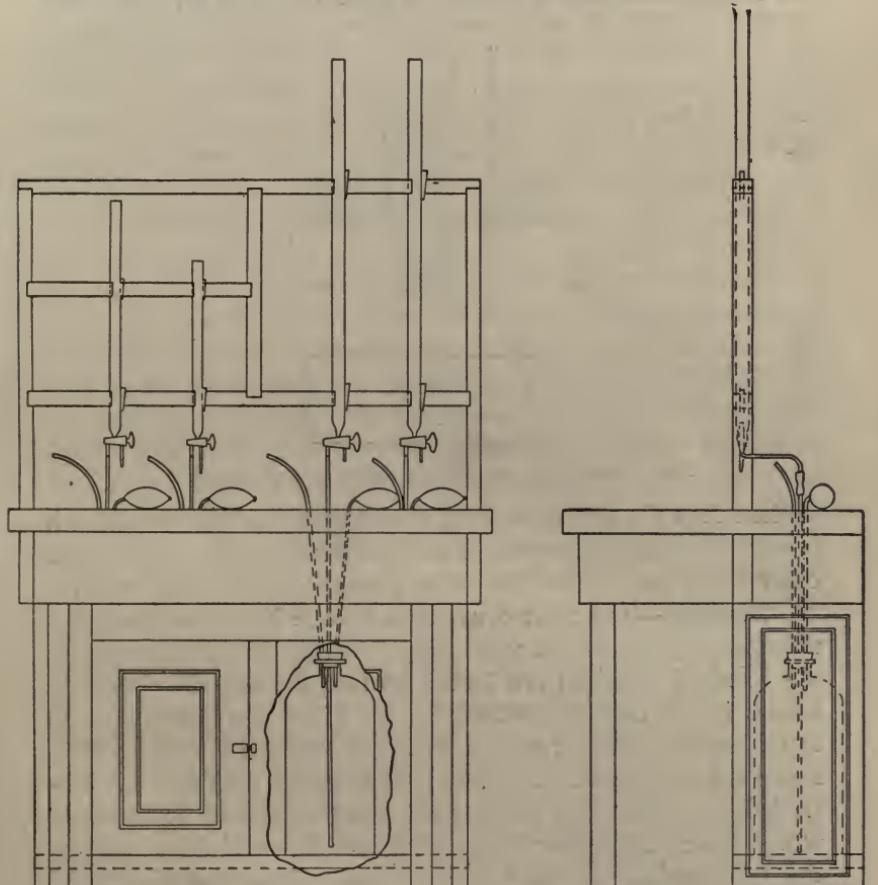


Fig 25.—Burette Table.

burette holder illustrated is merely a wooden frame. It is of the form shown, having holes bored through where the burettes are to go. The burettes themselves are held in place by a wedge, as illustrated in Fig. 26. In place of the frame shown, iron rods may be passed through holes in the table and fastened to the upper framework of the cupboard. The burette is then held in place by the ordinary burette clamps, over the jaws of which should be slipped a piece of rubber tubing to give them a firm grip. Or, since this tubing is apt to rot and stick to the burette, a piece of felt may be glued inside the jaws for this burette. Since burettes are always held in an upright position, the clamps with strong springs to close the jaws are more con-

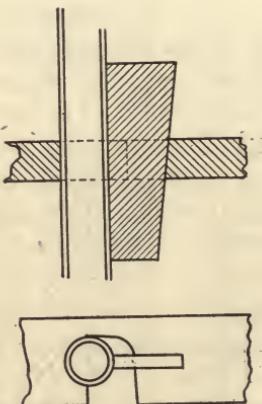


Fig. 26.—Method of Holding Burette in the Frame.

venient than the adjustable ones closed by a thumbscrew, since it is easier to get the burettes in and out for cleaning, etc. On the other hand, they do not last as long because the springs break.

**Burettes.**—The most convenient burette is the one having a three way stop cock and side tube for filling. In the table shown, the standard solutions are forced up into the burettes from the bottles in the cupboard below, by use of an atomizer bulb. In using, enough solution should be forced into the burette to fill it above the zero point. The excess is then run back into the bottle below. The bottle holding the solution should be provided with a three hole rubber stopper. Through one of these stoppers the tube leading to the burette passes. This tube should, of course, reach nearly to the bottom of the bottle. The second and

third holes are provided with short pieces of tubing reaching just inside the bottle. One of these tubes is attached to the atomizer bulb and the other to a piece of rubber tubing passing through a hole in the table top and extending about three or four inches above the latter. Instead of a three-hole rubber stopper, a two-hole one may be used and a Y-tube inserted in one of the holes. The atomizer bulb is then to be attached to one of the branches of the "Y" and the rubber tube to the other.

In forcing the solution up, the stop cock of the burette is turned to make connection with the bottle, the rubber tubing is pinched with the left hand and the atomizer bulb is worked with the right. When the solution reaches a certain point in the burette, determined by practice (usually the 25 or 30 c. c. mark), the pressure in the bottle is sufficient to fill the burette. At this point, the right hand is withdrawn from the atomizer bulb and placed on the stop cock. As soon as the solution passes the zero point, the connection between the bottle and the burette is cut off by the stop cock. All this time the rubber tube has been kept tightly closed by pinching between the thumb and forefinger of the left hand. This is now released and the pressure in the bottle relieved. The excess of solution in the burette can then be run back into the bottle. This same form of burette can also be obtained in a very convenient form, with an automatic zero point and an overflow reservoir similar to that of the pipette illustrated in Fig. 29, from Eimer & Amend, 18th St. and 3d Ave., New York. These latter are very useful, as they require no adjusting to the zero point when filling and consequently save time.

Instead of forcing the solution up from a bottle in a cupboard below, if permanganate, bichromate, iodine, and other solutions attacking rubber tubing or decomposed by the light are not used, a shelf may be placed on the wall back of the table and the solutions run down into the bottle. If, however, standard solutions attacking rubber are used, the cupboard below the table is the thing, since in the shelf arrangement the solution is in contact with the rubber tube used to join the burette tube below to the tube leading from the bottle; hence it soon eats a hole in the latter. If this happens at night, the whole bottle of standard solution will probably leak out, and an eight-liter bottle of permanganate would make a mess, in any event; while, if the laboratory was located on the second floor, it would probably damage

the ceiling of the room below. Where the solutions do not attack rubber it is, of course, more convenient to have the bottles on a shelf and run solutions down into the burettes by a siphon tube. The shelf, however, must be placed above the zero point of the burettes, in order to bring the solutions to the zero point when the bottles are nearly empty. When standard solutions are decomposed by light, they may be kept in bottles painted black with asphalt paint, or the bottles may be provided with a hood or bag cover, of heavy black cloth. When standard alkali and other solutions attacking glass are used, the burette should be of the old style Mohr's form, with rubber connections and attachments for filling from the side.

**Automatic Zero Burette.**—The ordinary form of burette may

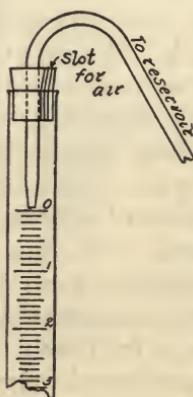


Fig. 27.—Burette with Automatic Zero Point Filler.

be made to answer as a zero point burette by the arrangement shown in Fig. 27. This consists in placing the top of the bottle holding the standard solution below the zero point of the burette. The tube leading from the former to the latter is then run down into the burette, level with, or a little above the zero point. This arrangement will siphon off the excess of solution in the burette, to the zero point, after a few adjustments and trials of the tube in different positions. The writer has at times experienced more or less trouble with this form of burette from the drop which collects on the end of the tube falling into the burette. This may be obviated by having the siphon tube drain into the bottle as much as possible and to this end it should be bent as shown in the

cut. The end leading into the burette should also be drawn very slightly towards a point. This latter will hold the drop. It is usually hard to get a rubber stopper small enough to go into the tube and with a large enough hole to admit the tubing, so as all that is needed is something to hold the tube in position an ordinary cork can be used. A few V-shaped grooves should be cut lengthwise down its sides to admit air in and out of the burette.

**Appliances to Aid in Reading the Burette.**—For aiding the operator in reading the burette, various appliances have been suggested. For colored solutions such as permanganate, nothing is needed, but for colorless ones floats are sometimes used. A new burette, designed by Schellbach, has recently been placed on the market and may be obtained from a number of dealers in chemical apparatus. This burette has a blue enameled line, with a white enameled background, running lengthwise down the burette. At the level of the solution, this line presents the appearance of an X and the level is taken as the point where the two V's come together.

Burettes having a white enameled background for use with colored solutions can also be obtained. A card having a piece of black paper pasted across its lower half, or having this part blackened, will aid in reading colorless solutions in an ordinary clear glass burette. If held with the line of division between the black and white, about an eighth of an inch below the surface of the liquid, and the eye brought on a level with it, the meniscus can then be seen by transmitted light, bounded below by a sharply defined black line.

**Caps for Burettes.**—If the top of the burette is open it should be closed by a small-glass cap, slipping loosely over it. One can be easily made from a test tube, as follows: Select a tube which slides over the burette top and mark it about one and a half inches from the closed end with a sharp file. Wrap two strips of wet filter paper around the tube, one a little above and one a little below the mark and about one-eighth inch apart. Direct the point of a small blowpipe flame against this opening between the two strips and revolve the tube. A crack will start and follow the flame around the tube. This is a simple way of cutting glass cylinders or tubes, and a five-pound bottle may be turned into a waste jar for the burette table in the same way; scratching it, wrapping wet newspaper around it, and cracking it with a flame.

The writer has found this way much more satisfactory than the methods usually recommended for the purpose.

**Portable Burettes and Solutions.**—Burettes mounted on the bottles containing the solutions with which they are to be filled are sometimes used and will be found useful when only a few titrations are to be made and these few not very often. They are usually fitted with automatic zero point. The forms designed by Squibb and Knopler are described in almost every catalogue of chemical apparatus. The great disadvantage of these arrangements is the small size of the bottles, which is usually two liters

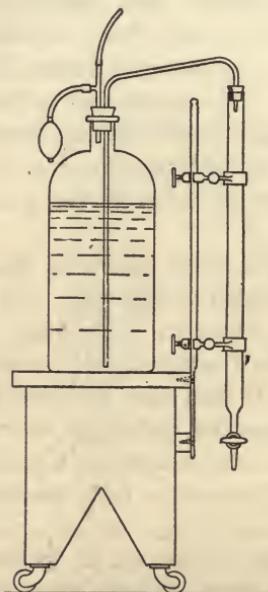


Fig. 28.—Burette and Solution on Movable Stand.

capacity, and of the burette, which is never larger than 50 c. c. If made larger the bottle is too heavy to move about, and the burette too long.

When space is limited, a burette and bottle mounted on a stand provided with rollers will prove handy, as it can be pushed back out of the way when not needed. Fig. 28 shows such an apparatus. Its construction is evident from the illustration. It consists of a stand, mounted on rollers, to the front of which is fastened an iron rod, two feet long and  $\frac{1}{4}$  or 5-16 inch in diameter. The lower end of the rod is flattened and through the flat-

tened end are bored two small holes. Screws through these holes fasten the rod to the stand. The burette is then clamped to the rod with jaw clamps and the solution is forced up either by a bulb or suction. The tube leading from the bottle to the burette is fixed to siphon off the excess to zero as described previously. The bottle should be kept in place on the stand by three small blocks nailed on top of the latter close to the bottle; for, if the bottle slides around, it will break the tube leading to the burette. Of course, any form of burette, either with or without automatic zero point, may be attached to the stand, and if the permanganate, bichromate, etc., are to be used with the apparatus, the bottle should be painted black, etc.

**Pipettes.**—For rapidly delivering known volumes of solutions the automatic pipette, shown in Fig. 29, will be found very useful. The bottle acting as a reservoir should be placed upon a shelf and the solution run down into the pipette. Since these pipettes are light compared with burettes, they can be joined directly on to the siphon tube leading from the reservoir by fusion and used for solutions, such as ammonium molybdate in nitric acid, which attack rubber rapidly. The siphon tube should be made of heavy walled glass tubing so as to resist breakage. The pipette may be fastened to a stand and the stand may be mounted on rollers, or not. Or the pipette may be mounted on the frame of the burette table. Before fastening the pipette to the stand or frame, the bottle should be placed on the shelf and moved about until the pipette is against its support. The pipette may then be fastened to the support by wire or a narrow thin brass band or spring.

Caustic soda and other solutions which absorb carbon dioxide should be protected from the air, or the air entering the bottle should be freed from the carbon dioxide by passing it over an absorbent. One of the simplest methods is to place a layer of kerosene on top of the solution, effectually protecting it from the air. Another method is to pass the air through a tube containing soda lime, which absorbs carbon dioxide. The soda lime tube may be stuck through one hole of a doubly perforated stopper, the siphon tube passing through the other.

Occasionally burettes will not run clean and small drops will adhere to their sides causing the readings to be too low. To remedy this, allow a weak solution of chromic acid to stand in the

burette for several hours. To prepare this solution, add about 25 grams of potassium bichromate to 150 c. c. of water and 15 c. c. of concentrated sulphuric acid. This solution will not attack the rubber tube of a Mohr's burette. Vaseline is probably as good a lubricant as can be found for the burette cocks.

**Indicator Bottles, Spot Plates, Etc.**—Indicators showing the end point of titrations should be kept in bottles provided with droppers. Any catalogue of chemical supplies will show several different forms, of which Schuster's, provided with a ground glass

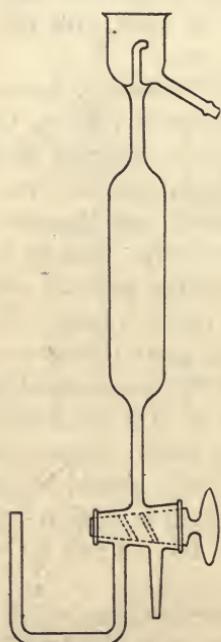


Fig. 29.—Automatic Pipette.

stopper, will be found convenient. A bottle through the cork of which a medicine dropper is inserted may also be used; or, in place of the rubber-bulb dropper, a piece of short glass tubing (5 in.) may be drawn out to a point at one end and blown into a bulb at the other. The warmth of the fingers placed on the bulb causes the air in the latter to expand, forcing a drop or two of the indicator out of the other end.

Spot plates of porcelain will be needed where outside indicators are used. These are glazed and may be purchased either

with or without cavities. These latter plates are the most convenient, as the drops are held in depressions and do not run together. A plate  $5\frac{1}{8} \times 7$  inches, with 30 cavities, will be found a good size for bichromate and ferrocyanide titrations. A spot plate may be made by pouring melted paraffine into a dinner plate and allowing it to cool. This plate may be easily cleaned by washing off with a stream of hot water from a wash bottle.

Porcelain plates are also used upon which to set beakers, flasks, etc., in order to observe the color changes during titration. Flat, square plates may be purchased for this purpose, or dinner plates or saucers may be used. Where titrations are made at night, the following device is said to be useful: A hole 6 inches square is cut in the table just below the burette, a ground glass plate is fitted over this and under the glass is placed an incandescent lamp with a white reflector.

Where solutions have to be titrated while boiling, a small electric stove or hot plate is said to be exceedingly convenient.

## CHAPTER VIII.

### BALANCE SUPPORT, BALANCE AND ACCESSORIES.

**Brick Pier Support.**—It is essential that the balance, used for analytical work, should be mounted on some firm support; as, not only does the accuracy of the weighings depend upon the freedom of the instrument from vibration and jar, but also the life of the balance itself. It is equally important, also, from the point of rapidity, since any disturbance of the swings makes a new trial necessary. The author has seen balances mounted so poorly, that walking across the floor, while the beam was oscillating, would cause a perceptible tremor of the pointer, and the slamming of a door, anywhere in the building, during the "swingings," was enough to throw the apparatus out of adjustment. The most satisfactory support for the balance is, of course, a masonry pier from the ground. The usual form consists of two brick piers, about two feet part and a brick and a half thick, on which rests a slate or stone slab. Almost every college laboratory has its balances so mounted, and, in some instances, the piers run up from the ground to the second and third floors. Concrete may, of course, be used in place of the brick piers. The stone slab is usually placed high enough from the floor, in the college laboratory, to permit of the student standing while weighing. In the mill laboratory, however, it will be found much better not to make the height so great; and to do the weighing while sitting down. In technical laboratories it is usual to weigh a large number of samples or crucibles at once, so that the operator would find it very fatiguing to stand throughout the operation. Where the laboratory is located on the first floor of the building, the mounting of the balance is comparatively simple, as all it will be necessary to do is to erect a couple of brick or concrete piers and to lay a slab of slate across these. The piers need not extend down into the ground for more than a foot, nor is it necessary to make them thicker than eight or ten inches. The concrete for the piers is made of one part Portland cement, three of sand and from four to six of broken stone or gravel. It should be tamped into the

wooden moulds. In place of the slate slab and piers, the all concrete table, Fig. 31, described a little further on, may be used.

Where a cellar comes below the laboratory it will probably be better to build one solid piece of masonry,  $24 \times 18$  inches, from the ground up, and bolt to this a slate slab,  $28 \times 28$  inches; so that it projects ten inches in front of the pier. Or else a pier  $30 \times 18$  inches may be built from the ground to the floor, and, on either end of this, two narrow piers,  $18 \times 6$  inches erected. A slab of slate,  $36 \times 20$  inches, is then to be laid over these. Slate slabs for this purpose need not be thicker than  $1\frac{1}{2}$  to 2 inches. Where slate or stone slabs can not be obtained, a top of two-inch seasoned and neatly dressed board may be made to serve instead. As

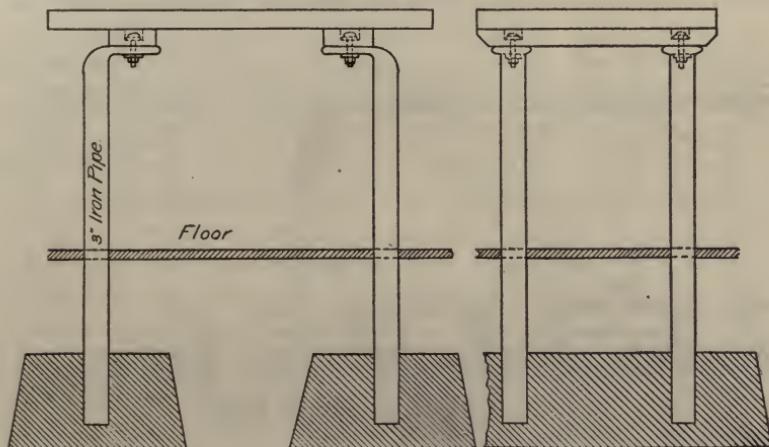


Fig. 30.—Iron Column Support for Balance.

the board is not heavy enough to stay down of its own weight, it must be bolted to the pier. This may be done by bolting the battens to the pier and then screwing the top to the former. The screws should be sunk about an inch below the level of the surface and the holes then stopped up with a round peg and glue, and the whole smoothed off with a plane and sandpaper. Or the top may be bolted directly on to the pier, and the bolts so arranged as to come out of the way, under the balance.

**Iron Column Support.**—Figure 30 shows a simple way of mounting a balance solidly. It consists of a wooden top fastened to two battens, which are in turn bolted to four iron columns, which are imbedded in concrete in the ground. The columns are

made from 2 or 3-inch wrought iron pipe by flattening and bending. Any blacksmith can make them. The concrete piers are only large enough to give the necessary stability and need not come above the ground. If made  $12 \times 18 \times 36$  inches, they will be of ample size. The battens are first bolted to the columns, the heads of the bolts being sunk below the upper surface of the battens; the latter being trued up so as to make the table top level, by the use of metal liners. The top, which should be made of well seasoned 2-inch lumber, is then fastened to the battens by means of screws, from below. In order to get the columns in proper position in the concrete, it will be found simplest to make a rough frame and bolt the columns to this, so that their tops are all on a level, and then to pour the concrete into the moulds and

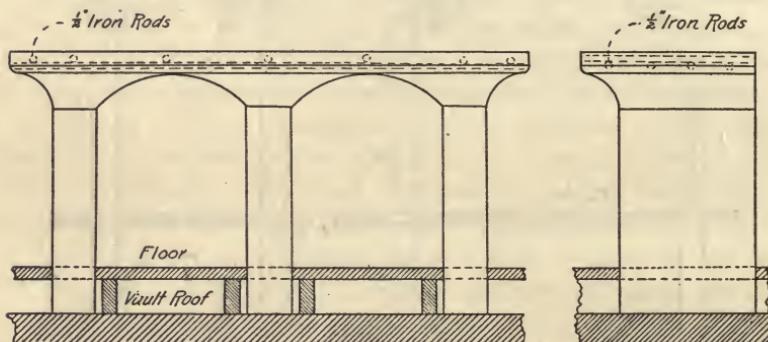


Fig. 31.—All Concrete Balance Support.

around the lower ends of the pipes. The table top may be filled and varnished and the pipe columns should be painted with aluminum paint—such as is used for radiators, etc.—or with black asphalt varnish. Where a plank top is used for a balance table, particularly if this is made of white pine or other soft wood, the legs of the balance case are apt to sink into the wood, and it is therefore well to rest the legs upon small pieces of metal, such as one-cent pieces.

**Solid Concrete Support.**—Figure 31 shows the arrangement for mounting the balances in the laboratories of the Dexter Portland Cement Company. The laboratories are on the second floor, and the balance room is built above a large, fireproof, concrete vault, used for the storage of the books, papers, etc., of the office below. On the roof of this vault a concrete table for the bal-

ances has been built. Its construction is evident from the illustration. The concrete was made of a mixture of one part Portland cement and four parts limestone screenings. The upper surface of the table was trowelled, while the concrete was still wet, until it presented a smooth glassy surface. A wooden form was, of course, first made, iron rods were inserted as shown in the cut, and the concrete was poured into this form, and tamped and trowelled as usual. The wooden forms should be fastened together with screws to avoid breaking off the corners and edges of the table in removing the boards. This concrete table presents a pleasing appearance to the eye and is satisfactory in every way.

**Shelf Support.**—The rigid mounting of balances in laboratories located on the upper floors of factories and other buildings often presents a problem difficult of solution. If the building is of brick or stone, the best plan will be to rest the balance on a shelf supported from the outer wall by iron or heavy wooden brackets, bolted fast to the former. In factory or mill buildings, much of the shafting is supported by the girders and beams of the floor above, and consequently the latter trembles more or less all the time when the machinery is in operation. Where the laboratory is on such a floor, it is almost an impossibility to make a weighing when the balance merely rests on a table. In such cases, if it is impossible to bolt a shelf on to another wall, the following arrangement may be resorted to, and will deaden the vibrations to a certain extent. First, a very heavy wooden table is made and on this is placed a slab of slate, stone or even metal, resting on six or more solid rubber balls an inch and a half or two inches in diameter. The heavy slab takes up much of the jar. The addition of another slate slab, resting on rubber balls, which in turn rest on the first slab, will still further lessen the jar. As the weight of the slab flattens the rubber balls somewhat, it is not necessary to scoop out depressions in the table or slab in which they may rest. When a table is made use of to support the balance it should be made heavy and substantial.

**Location of the Balance Support.**—The balance table or support should be placed in a good light but not where the sun will ever shine directly upon it. If it must be placed by a window which would permit the sunlight to fall upon it, the former should be provided with a screen or awning to prevent this.

**Balance Support Bench.**—Figure 32 shows a small bench or seat for use with the balance table. Its form is evident from the cut. It is made of  $\frac{1}{8}$ -inch boards and its dimensions are—width, 14 inches; length, 16 inches; height, 18 inches. The small hand hole in the top of the bench is to carry it about by. A table made similar to this only larger and of much heavier lumber will also make a good balance table. In this case the dimensions should be about as follows:—width, 20 inches; length, 30 inches; height, 30 inches.

**The Balance.**—It is perhaps well here to say something about the balances themselves. Two general types of balances are on the market in this country. In the first of these types, shown in Fig. 33, the beam itself is graduated to receive the rider

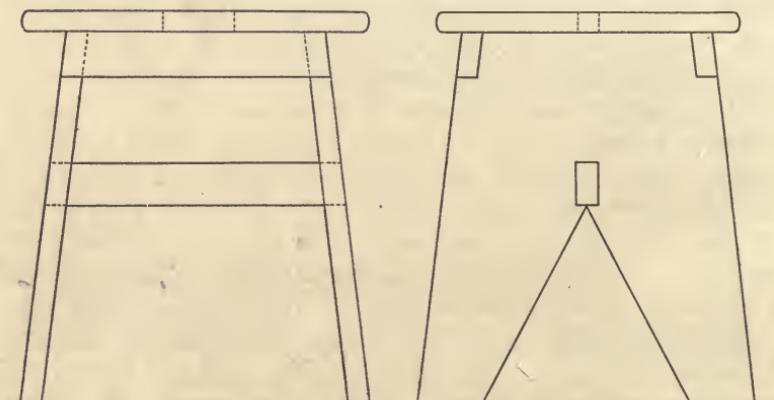


Fig. 32.—Laboratory Bench or Stool.

directly, and is made in the form of a very much flattened isosceles triangle with the apex pointing downward. The balances manufactured by Becker's Sons (Rotterdam), Christian Becker (New York), H. Kohlbush (New York), Henry Troemner (Philadelphia), and Wm. Ainsworth (Denver), all have the characteristics of this type. In the second type, shown in Fig. 34, the rider moves upon a scale separate from and fastened to the beam, and the latter is in the form of an isosceles triangle, much more acute than in the former type, and with the apex of the triangle pointing up. Sartorius and Staudinger, Arthur H. Thomas Co., Agents, Philadelphia, balances are representative of this type. Balances of first type are the more used, however, in mill, furnace, and smelter laboratories, in this country. Enough money



Fig. 33.—Analytical Balance—Becker's Sons.



Fig. 34.—Analytical Balance—Staudinger.

should always be put in a balance to get a good one. This does not mean a showy one, but one which will do accurate work under the trying conditions of technical analysis. The short beam balances will be found accurate enough for technical work and they are much more rapid. Nothing is gained by having an analytical balance which will be sensitive to less than 0.1 milligram with a load of 100 grams, since this is a degree of accuracy seldom reached by the other operations of a technical analysis. For assay work, however, button balances, sensitive to 0.01 milligram are desirable. A balance with an aluminum beam is to be preferred to one made of brass, since the lighter the beam the greater sensibility the balance possesses. The knife edges should be preferably of agate, and the planes always of agate.

The arrangement of the arrest of beam and pans should be such that when the balance is at rest, the knife edges and planes are not in contact; and if the pan arrest moves in the same arc as the beam itself it is a point in favor of the balance, as the dulling of the knife edges by dragging them across the planes, which happens when the weight in one pan is two or three grams heavier than the other, is avoided. The balance should be preferably mounted on a glass plate.

**Assay Balances.**—These are of two kinds—button balances and pulp balances. The former are used for weighing the buttons of gold and silver obtained by cupellation and the latter are for weighing the samples of ore. Button balances are made much more sensitive than analytical balances and are intended for weighing only a few milligrams. The pans are much smaller, are concave and removable and the beam is much lighter. They are now usually made with the rider attachment. Troemner and Ainsworth both make assay balances sensible to 1-500 of a milligram. For ordinary purposes a balance sensible to 1-50 of a milligram will be sufficient. All the bearings of this, however, should be of agate. Small platinum weights, 1 milligram and up, are used. F. W. Thompson, of Denver, Colo., makes an assay balance, in which all of the small weights (72 milligrams) are in the form of riders. These are so suspended from a carrier that they may any or all of them be dropped over a projecting arm on the wire suspending the right hand pan. This does away with handling the small weights.

Any balance sensible to two or three milligrams and having a

capacity of two or three hundred grams will answer as a pulp balance.

**Weights.**—The weights must, of course, be accurate, and a new balance and weights should always be tested. It is not necessary for ordinary technical analysis that the arms of the balance shall be of equal length, nor that the weights should be compared with the standards in Washington, but the balance should be sensitive to at least 0.2 milligrams with a load of 50 grams on each pan, and still better, to 0.1 milligram with a load of 200 grams. The latter requirement is enough for all analytical purposes. It is also essential that the weights should be relatively correct with regard to each other; that is, the 10-gram weight must be equivalent to exactly ten times the weight of the 1-gram piece, etc.

**Balances for Rough Weighing.**—In every laboratory there will be needed a balance capable of weighing quickly and roughly 200 or 300 grams of salts to be used in making up solutions. The balances with the pans above the beam will be found the most convenient of these. The torsion balances are made to cover a wide range of work:—for example these can be obtained with pans 4 inches in diameter, a capacity of 500 grams and a sensibility of 5 milligrams, or with 6-inch pans, a capacity of 5 kilograms and a sensibility of  $\frac{1}{2}$  gram. The latter may be obtained with beam graduated to  $\frac{1}{2}$  gram. The “Harvard Trip Balance” is also convenient. This has 6-inch porcelain plates in place of pans and a graduated beam (1-10 gram to 10 grams). It is sensible to 1-10 gram and has a capacity of 1 kilogram. “Troemner’s New Laboratory Scale” is also well suited to this work. It has 6-inch pans, a capacity of 200 grams and is sensitive to 1-20 gram. The weights are kept on a projecting shelf at the base of the balance. The Harvard and torsion balances will need a set of cheap weights ranging from 200 grams to 1 gram.

**Pans, Etc.**—In most technical laboratories, it is usual to weigh out an exact amount of a substance; and for this purpose counterpoised watch-glasses are usually used. That is, two watch-glasses balanced against each other. These can be bought of any dealer in balances or chemical supplies; but, if the chemist desires, he can make a pair himself from ordinary watch-glasses, by selecting from his stock the two which agree most closely in weight, and grinding and filing until they balance. If they are

to be left on the pans when crucibles are weighed, the final adjustment of this may be done with the aid of the screws on the end of the balance beam.

Where a sample has to be transferred to a flask, it is necessary, if a watch-glass is used, to brush the sample from this on to a piece of glazed paper, and then from this in turn to the flask. To avoid the double operation and chance of loss of the substances, it will be found best to substitute a pair of counterpoised celluloid pans for the watch-glasses. These are made by cutting out squares of thin clear celluloid, with rounded corners, large enough to completely cover the balance pans. These can be curved between the fingers, to fit the mouth of the flask, and the sample brushed from them directly into the flask. Aluminum foil may also be used for these pans. If a watch-glass is used it should completely cover the pan, so as to avoid danger of the material dropping on the pan and being included in the weight but not in the sample.

To transfer the sample to the pan and remove the excess, a small spatula is used. This should be ground down on an emery wheel or grind-stone so it tapers to a rounded point of about  $\frac{1}{8}$ -inch width. When it is to be used for iron and steel analysis, it should be magnetized by rubbing across the poles of a bar magnet or dynamo. In weighing pig iron samples, however, care should be used not to do anything more than get the final adjustment to the weight by the use of its magnetic properties, as the non-magnetic particles of the sample contain a larger proportion of the metalloids than do the magnetic ones, and an undue proportion of the former may be left.

To brush the sample from the watch-glass or celluloid pans, use a flat sable or camel's hair brush,  $\frac{3}{4}$  to 1 inch wide. To transfer the sample, rap the glass or pan gently with the brush handle, until most of the material falls into the dish or flask, and then brush in the few particles which remain attached.

In water analysis, alkali determinations, etc., where a residue of deliquescent salts, left from evaporation, has to be weighed in a platinum dish, it will be found almost an impossibility to get an exact weight owing to the absorption of moisture from the air by the contents of the dish. A useful adjunct of the balance will therefore be an aluminum box, with cover, large enough to hold the dish. Such boxes are sold for holding soap, salves, etc., and

while the covers do not fit tight enough to make them air-tight, they do fit tight enough to keep the moisture of the air from coming in contact with the contents of the dish during the short time necessary to make a weighing. The box may be counterpoised by a small piece of brass, filed down to balance it; or the dish and box may be weighed together. In use the box should be dried in the desiccator while the dish is cooling. When the dish and contents are cool, they are to be placed in the box, the cover fitted on and the weight immediately taken. If an aluminum box can not be obtained a seamless tin box such as is used for salve or blacking may be employed. It is much heavier than the aluminum box, however, and in large sizes so much so as to prevent it being used.

Where hygroscopic substances have to be weighed, weighing bottles will be needed. These may be purchased with light blown glass stoppers and in a variety of forms, preferably with flat bottoms and straight sides. When samples of ore, etc., are to be weighed, a small bottle 25 mm. in diameter and 40 mm. high will be found convenient. If the sides of the bottle are straight the sample can be brushed from the bottle with a round camel's hair brush. These little bottles can also be obtained in a squat form, as wide as 70 mm. and as low as 30 mm. Such weighing bottles are well adapted to weighing the residues from evaporation and drying, both operations being conducted in them. They should be cooled in the desiccator with stopper out. In weighing the bottles the stopper should always be removed for a second, just before weighing, to allow the pressure to equalize. Counterpoised filter papers are usually supposed to be weighed between watch-glasses held together with clips. A weighing bottle 70 mm. high and 30 mm. in diameter will be found much better, as it is lighter. This size will take a 11 cm. filter paper. Hygroscopic precipitates may be weighed, crucible and all, in such a bottle. To give an idea of the lightness of these little blown glass-stoppered weighing bottles, one which the author has, measuring 50 x 30 mm. and holding 30 c. c. weighs only 18 grams.

## CHAPTER IX.

### HEATING APPLIANCES.

**General Considerations.**—If the laboratory is so located that coal gas or natural gas is accessible, the problem of heating hot plates and making ignitions simply resolves itself into the use of gas stoves and burners of the simplest types, such as are familiar to every student of chemistry. When, however, the laboratory is located, as most small ones are, at some mine, smelter or furnace, no such convenient means is at hand and something must be substituted for city gas. Where fire assays are made, the chemist will of course find the muffle of the assay furnace all that is needed for burning off his filter papers and igniting his precipitates; and oil stoves or stoves heated by wood or coal may be used to evaporate solutions, boil water, etc. In the east, in the laboratories of blast furnaces, cement mills and various and sundry manufactories, there are of course, no fire assays made and the heating here is usually done with gasolene.

This is used in one or two ways; either the gasolene is burned directly in a suitable burner or lamp, or else it is vaporized by a current of air and the mixture is burned in some form of Bunsen burner just as if it was coal gas. The latter method is the most convenient one but requires the use of a generator to vaporize the gasolene. The latter is somewhat expensive if purchased, but in many cases may be constructed by the mechanics employed at most manufacturing plants at a comparatively small cost. The lamps for burning gasolene directly are troublesome and not altogether free from danger, unless carefully handled. They require frequent attention, and usually have to be filled at least once a day.

**Gasolene Lamps.**—Of these lamps the best known is that of Dangler, which is shown in Fig. 35. In order to start the lamp the reservoir, *A*, is filled with gasolene, the bulb, *B*, is compressed a few times to put the gasolene under pressure and the valve, *C*, is opened enough to let a little gasolene into the pan *D*; this is then ignited with a match and when the burner has been heated, the gasolene is turned on, vaporized by the hot burner and ignited

at *E*. These lamps give a flame which can be regulated from the size of that of an ordinary Bunsen burner to a powerful blast. They cost between \$5.00 and \$6.00, can be purchased from any supply house, and are very similar to the torches used by painters, plumbers, etc.

Figure 36 shows the burner manufactured by The Hoskins

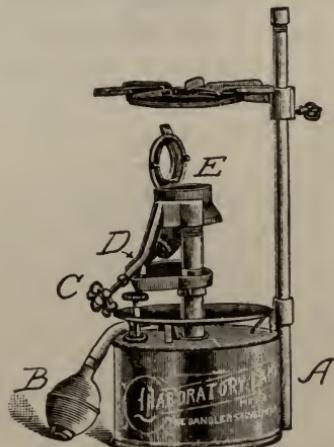


Fig. 35.—Dangler's Gasolene Lamp.

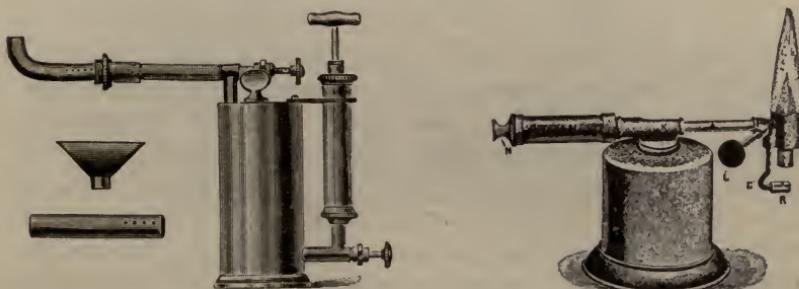


Fig. 36.—Hoskins' Gasolene Lamp.

Fig. 37.—The Jewel Gasolene Lamp.

Company, 93 Erie St., Chicago. This burner is an improvement on the Dangler burner. It is more portable and the substitution of the metal pump for the rubber bulb, which wears out rapidly, is a good feature. The flame is under perfect control and the lamp is substantially put up. The extra tubes are for a fish tail flame, for bending glass, and one horizontally directed.

Figure 37 shows the Jewel gasolene lamp for sale by E. H. Sargent & Co., Chicago. This is a very small, new type of gaso-

lene burner which meets all the purposes of a Bunsen burner. It generates its own gas and is practically automatic in operation. The tank or reservoir which forms the main body of the lamp holds about one-half pint of gasoline. Air pressure for operating this torch is obtained by means of a small force pump contained in the handle. The flame is adjustable in size from almost nothing to 5 to 6 inches, and will burn about  $1\frac{1}{4}$  hours at full blast from one filling of the reservoir. The ease with which this burner is lighted is a most desirable feature. The heat of one or two matches is all that is necessary to generate the burner. Figure 38 shows the generator being heated preparatory to lighting the burner.

**Stoves.**—For evaporating solutions, etc., a gasolene stove such as is used in kitchens will be found a necessary adjunct to the gasolene lamps. Or a kerosene stove may be used. These

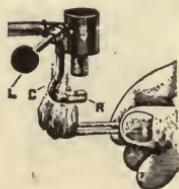


Fig. 38.—Lighting the Jewel Lamp.

latter can be obtained either with or without wicks, the wickless "blue flame" ones being the best, although a little more expensive than the older form. These stoves are usually mounted on an iron frame and are intended to stand directly on the floor. It will be found most convenient, however, to raise the stove six or more inches and to build a hood over it. The stove should be covered with a cast iron plate and, if many samples have to be dried or moisture determinations made, the ovens sold with these stoves for baking purposes may be used.

In place of the oil or gasolene stove, an ordinary wood or coal cooking stove may be used, or a hot plate may be rigged up over a grate, so as to be heated by wood or coal. Neither of these schemes are desirable, however, except where kerosene and gasoline can not be obtained. Mr. Herbert Haas describes such an arrangement<sup>1</sup> designed by him for the laboratory of a pyrite smelter in California, as follows:

<sup>1</sup>Electrochemical Industry, III., 3, 101.

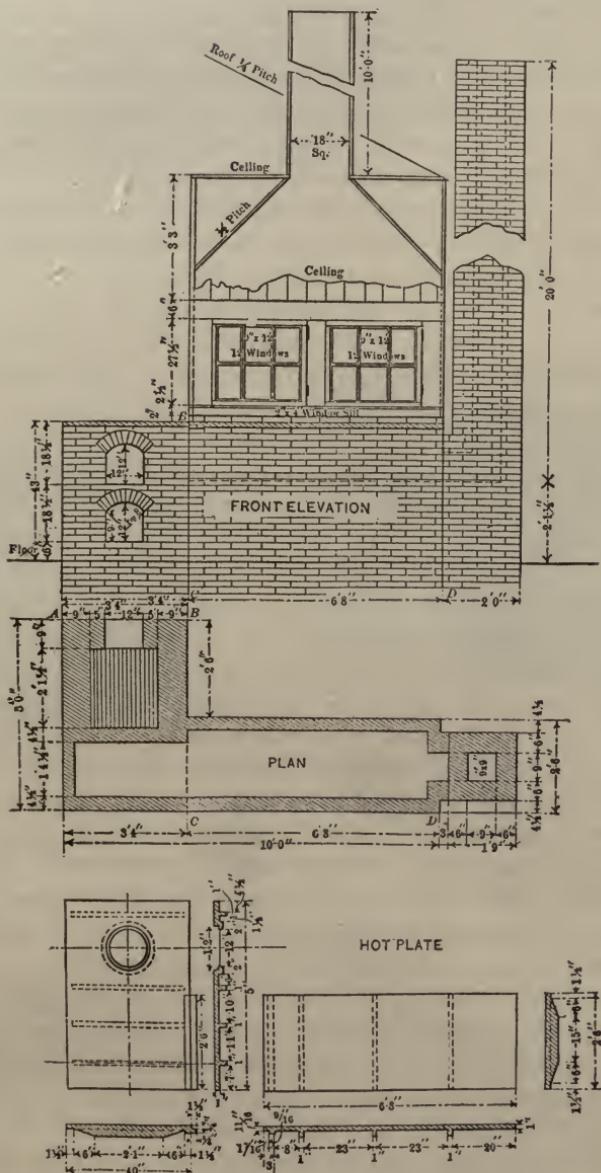


Fig. 39.—Laboratory Hearth Using Wood for Fuel—Haas.

"The hearth, Fig. 39, rests on the ground and the flue from it is filled with ashes and earth to within 18 inches of the hot-plate. In front of the ash-pit, beneath floor, is a 12 x inch inch opening, which is closed with a piece of sheet iron luted on with clay. The accumulated ashes in the ash-pit are removed through this opening, thus avoiding their removal through the office. The ash-pit door, shown in the drawing, is used solely to regulate the draft. Old rails, preferably, are used as grate bars.

"The front elevation of the hearth is shown in the illustration, which includes also the elevation of the fireplace front, with the ash-pit door and the feed door. The lines *AB* and *CD*, explain the respective elevations. The walls of the hearth consist of one course of brick, excepting at the stack, which is of a course and a half, and the fire-box, which is of two courses. These walls support the hot-plate, having its upper surface 43 inches above the level of the floor. A detailed dimensioned drawing of the hot-plate is given in the lower part of the drawing. The plate is cast in two pieces, having a lap so that a tight joint may be obtained, and at given intervals ribs are cast as a safeguard against warping. A circular hole, over which the still is placed, is left in the plate. A portion of the hot-plate 2.5 feet by 6 feet 8 inches, is covered with a hood, which rests on one layer of bricks, except at the hottest parts, where there are two layers in order to protect the wood. The back side of the hood does not rest on bricks, but is separated from the plate by a 2-inch air space extending the entire length of 6 feet 8 inches. Access to the hot-plate is obtained through two windows, each having twelve lights of glass and hinge on the butts. The hood is tightly ceiled with tongue and groove lumber, and has an 18 x 18 inch wooden chimney, 10 feet high, to carry off the fumes. The temperature of the inside of the hood and hood chimney is sufficient to draw in fresh air constantly and thus improve the ventilation of the laboratory.

"The great advantage of the hot-plate is in its gradual decrease in temperature towards the chimney. The heating of solutions is generally started at the coolest place, and gradually continued toward the hottest part. The heat is diffused over a large area, and is not concentrated at one small spot, as is the case with a Bunsen burner; and the boiling over of solution is thus easily avoided at the expenditure of the least attention and care; this allows the chemist time in which to attend to other work. A

small, uncovered portion, 2.5 feet by 3 feet 4 inches, is reserved for operations which are preferably conducted in the open air.

"A still, placed over the circular hole described above, provides the laboratory with about 14 gallons of distilled water daily. The still now used is called the Cuprigraph Sanitary Still, No. 11, and when once regulated requires very little additional attention. The hearth thus serves two purposes, one to provide the laboratory with distilled water, and the other to give the chemist, at the same time, a very efficient way of heating solutions. The distillation of the water utilizes much of the heat, yet the hearth is necessarily wasteful, the flue being too short for economizing fuel. The weekly fuel consumption, however, is only from one-half to

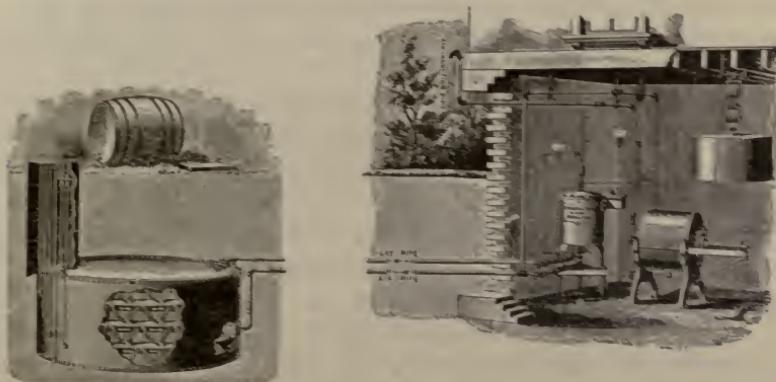


Fig. 40.—Springfield Gas Machine.

three-quarters of a cord of wood, which at \$4.00 a cord is equivalent to an operating expense of from 28 to 42 cents a day."

**Gas Machines.**—Of the machines for generating "gasolene gas" the "Springfield" is perhaps the best known and is manufactured by The Gilbert & Barker Manufacturing Co., 80 and 82 Fourth Ave., New York. The Springfield gas machine consists of a gas generator, which consists of a cylinder containing evaporating pans or chambers, a regulator for mixing the air and vapor in the proper proportions, and an automatic air-forcing apparatus.

The general plan of setting the apparatus, and the arrangement of connecting pipes, is shown in Fig. 40, which illustrates a Springfield gas machine, set up and connected, in every particular, in accordance with the latest rules of the New York Board of Fire Underwriters. The automatic air pump is here seen in

the cellar of the laboratory, and connected to it and running in the ground is the air-pipe conveying air from this instrument to the gas generator, located underground, and removed from the building thirty, fifty, one hundred feet, or more.

When the machine is in operation, the pump forces a current of air through the generators; here it becomes carburetted, thus forming a combustible gas that is returned to the cellar, where it passes through the regulator and, if too rich in gasoline, air is added to make the mixture about 15 per cent. gasoline vapor and 85 per cent. air. From the mixer the gas goes to the burners.

Gas is generated only as fast and in such quantities as is required for immediate consumption. The process is continuous while the burners are in use, but instantly stops when the lights are extinguished. One gallon of gasoline will make about one thousand cubic feet of gas.

In place of the air pump operated by a weight, one run by water can be substituted. This latter form is to be preferred, as it does not need any attention. It does not require any head of water to operate, and two gallons of water are said to be sufficient to run one burner one hour. Both forms of pump give a steady pressure. These gas machines give excellent service and very little trouble.

A machine which gives good results but which is not quite so convenient is described below. It is similar in the main to one designed for the laboratory of the Edison Portland Cement Co. by the author. The generator is shown in Fig. 41. It consists of an ordinary galvanized iron tank, such as is used in connection with kitchen ranges for holding hot water. A hole large enough to admit the hand is cut in the side of this tank at *B*, and two semi-circular pieces of light angle iron, bent to conform to the inside of the generator, are bolted inside as shown at *h*. The upper side of these angle irons are punched with holes, as illustrated in the small sectional drawing, and in these stout wires are fastened to make a sort of grid across the tank. Pieces of lamp wick, *d*, *d*, *d*, etc., long enough to reach to the bottom of the tank are hung down over these iron wires and the hole *B* is then covered by bolting on it a piece of metal. The joints of this, and also of all piping, are covered with solder, so as to prevent any possibility of a leak. The air pipe, *E*, and the gas pipe, *F*, and the fill pipe, *G*, should all have been screwed in place before hanging

the wick over the wires. The tank is now buried in the ground, The pipe, *F*, should of course run to the laboratory and connect with the gas jets. The fill pipe, *G*, should reach 8 or 10 inches above the ground and be closed by a cap containing a washer. The pipe, *E*, leads to the air pump to be described later. The generator is filled with gasolene to a height of about 18 inches from the bottom, and fresh gasolene added every day or so to make up for that used. The height to which the gasolene has risen in the generator may be ascertained by fastening a small test tube on to a piece of stout iron wire, and noticing at what point this comes up full, when lowered into the tank, through the fill pipe, *G*. (In putting in the wires care should be exercised to allow room between the wires and wicking, at this point, to lower the tester.)

For furnishing the air for this generator a number of devices may be used. If only three or four burners are used at one time and a head of water is at hand nothing will be found so convenient as the water blower described in Chapter V.. If more than this number of burners are to be used and plenty of water is at hand two or more of these blowers may be used, or a larger pipe or iron tank may be substituted for the pipe, *B* (Fig. 22), a larger over-flow pipe used and several aspirators screwed into this tank. One or more of these aspirators may be used as the amount of work done in the laboratory requires it.

In place of the water blower, if power is at hand, as is usually the case in most mill or furnace laboratories, the air may be supplied by a small Root or Crowell positive pressure blower, belted on to a shaft, in some convenient place about the mill. These blowers can be obtained from most dealers in laboratory supplies. Any means which will give a constant supply of air, at an unvarying pressure of about one pound per square inch, or even less, will answer the purpose of an air pump for this machine. The pressure of course must be low or the burners will be hard to light. Any change of pressure will also usually result in the flames dropping or being blown out.

This gas machine can be constructed with water blower for about \$25 or with the Root or Crowell blower for about \$40.00, depending very much on the amount of piping that has to be done.

Gasolene for these machines should be what is known as 88°

and should be purchased if possible in iron drums. A spigot may then be screwed into the drum and the latter set on its side, or a suitable rest, and the gasoline drawn as required for replenishing the generator. The gasoline should be kept in a small shed, away from other buildings.

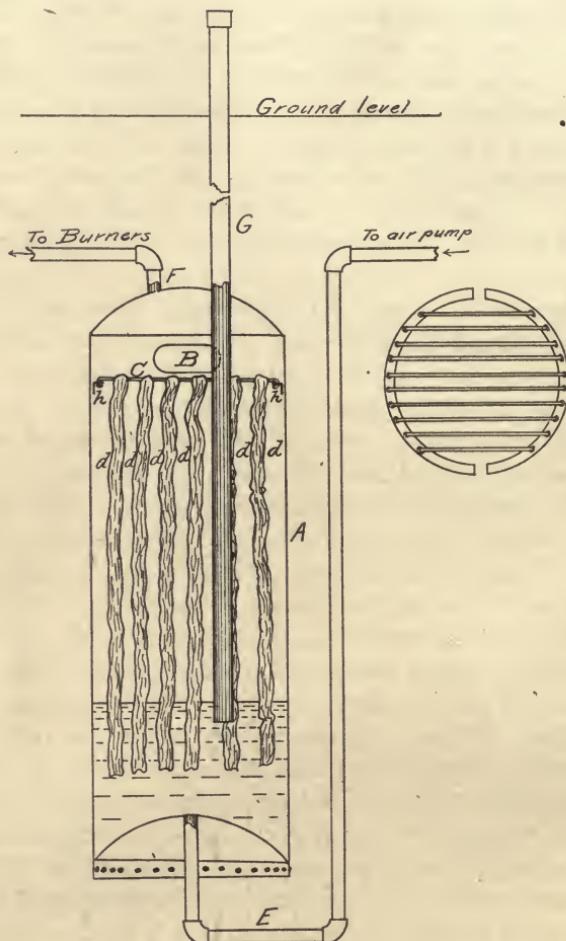


Fig. 41.—Simple Gas Machine.

**Acetylene.**—Acetylene gas is now used for laboratory purposes and quite a number of firms manufacture generators and burners especially suited to laboratory requirements. Acetylene gas gives off great heat when properly burned, but it requires special pattern Bunsens and stoves to burn it without loss. It is

stated by one chemist who uses acetylene that it does not cause the deterioration of platinum ware which other forms of gas do. It compares very favorably with gasoline gas in economy.

Where much work is done with crucible furnaces and high temperatures are needed, acetylene gas may be the best gas to install, as it is possible with it to produce temperatures higher than those obtainable with gasoline gas or even with coal-gas.

**Burners.**—Figures 42, 43 and 44 show forms of burners well suited to gasoline gas. The author has always found the form shown in Fig. 42 to answer very well, but the other two burners have the additional advantage that they do not light back and hence are safer.

Where burners are to be used for heating dishes, a crown



Fig. 42.

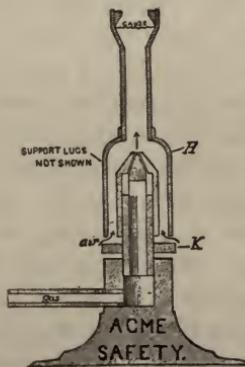
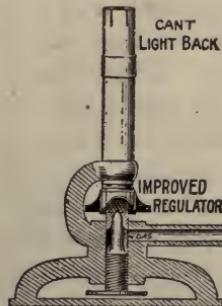
Fig. 43.  
Burners for Gasolene Gas.

Fig. 44.

or gauze top for the former distributes the heat more uniformly. For bending glass a wing top is needed. Stars, which slip over the tube of the burner and can be held at any height, are used to support chimneys of glass, sheet iron, or best mica. These chimneys keep the flame of the burner from being blown about by every draft. Julian has devised an adjustable support for dishes, platinum triangles, etc., which can be slipped over the tube of the burner and kept at any height by a thumbscrew. This does away with an independent support.

Porcelain burners are obtainable for use in hoods and other places where metal burners would be badly corroded. One of the neatest forms of these is Chaddock's. As this burner is provided with a chimney, on which to set a triangle, and a support for dishes, etc., no metal tripod or retort stand has to be used.

When uniform heat is desired, as in evaporating solutions, an argand burner with chimney may be used. If ordinary burners are used, place on top the gauze on which the dish is supported, a round piece of asbestos paper the size of a silver dollar and then place the dish on top of this and the burner directly under it. This spreads the heat, and the evaporation can be made to proceed evenly without ebullition. Teclue burners are very powerful burners and are usually used for heating large sand, air or water-baths. Burners with from 2 to 6 tubes, either arranged in clusters or in a row, are also used for the same purpose.

**Stoves and Hot Plates.**—The simplest hot plate is a sheet of boiler plate,  $\frac{1}{8}$  to  $\frac{1}{4}$  inch thick, resting on a tripod and heated by a Bunsen burner. An improvement on this is a plate resting on a Fletcher's burner. Since cast iron does not warp as bad as wrought iron a stove lid makes a good hot plate and may be used where only a little work is done, being heated by a Fletcher or a Teclue burner. Gas stoves may be procured either of chemical supply houses or else from local dealers in gas fixtures. The most convenient thing, however, is the regular hot plate sold by chemical supply houses and consisting of a gas stove having a polished cast steel top. The burners are arranged to give a uniform heat and they may be lit as needed so as to have one part of the plate hotter than the other. A hot plate  $14\frac{1}{2} \times 18\frac{1}{2}$  inches arranged for gasolene gas may be procured for \$12.00.

Sand-baths are convenient for heating dishes and other round-bottomed utensils, but they are dirty things at best and unsuited to the analytical laboratory.

**Water-Baths and Air-Baths.**—The water-bath is much used for evaporation and is made of both enameled ware and copper. They may be purchased with any number of holes, which are covered by concentric rings so that any sized dish may be set upon them. They may be heated by a Bunsen burner, or, if steam is run into the laboratory, they may be attached to this. Water-baths are also obtainable for keeping funnels and contents hot during filtering. Water-baths heated over a burner are liable from carelessness to boil dry. To guard against this, the constant level apparatus is used in some laboratories. This is shown in Fig. 45.

The bottle, *B*, is filled with water and closed with a single-

hole cork having in it a piece of short glass tubing, *d*. This is placed in the cup, *c*, of the water-bath, *A*. When the water in *A* falls below the end of the tube, *d*, air enters the bottle and allows a corresponding amount of water to flow out. As soon as the water in the cup rises above the end of the tube, no more water, of course, can run out of the bottle. This is a clumsy arrangement, however, and may be dispensed with, as a very little experience teaches the chemist how much water he will require in his bath to run it the time he needs it.

Air baths for drying materials are needed in all laboratories, and are used for determining moisture in samples and for drying ores, coal, etc., etc. They usually consist of a copper box provided with a hinged door in front and holes for the insertion of a

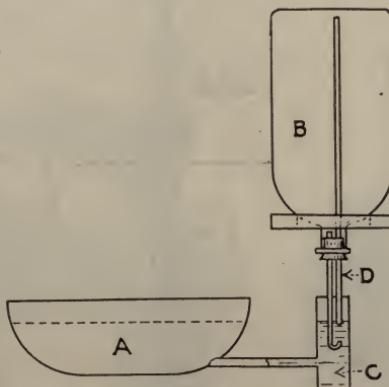


Fig. 45.—Constant Level for Water Baths.

thermometer and for the escape of water vapors in its top. The bath is provided with a false bottom, to prevent destruction of the real ore by the burner flame. The bath is heated by a Bunsen burner. It is necessary to control the temperature of the bath by a thermometer inserted through a cork in the opening at the top of the oven. The required temperature can be maintained by adjusting the stock cock of the gas supply. Gas regulators called "Thermostats" can be purchased from dealers in chemists' supplies, and, while they are liable to get out of order by becoming clogged up, they are nevertheless very convenient for keeping a constant temperature. As any checking of the gas supply is likely to cause a gasoline gas burner to strike back, it is best, where a thermostat is used with this, to have two burners, only one of

which is controlled by the thermostat. The other burner is turned low and the two burners are tilted so their tops are close together. If the burner attached to the thermostat goes out, as it probably will when the gas supply is checked, it will be lit by the other one.

Figure 46 shows a home-made thermostat. A piece of large bore tubing is blown into a small bulb, *A*, at one end and then about 2 inches from this bent into a U, *B*, as shown in the cut. This *U* is filled with mercury nearly up to the bulb. A piece

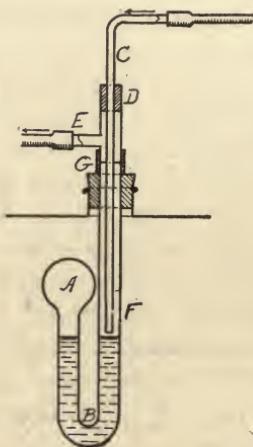


Fig. 46.—Thermostat.

of thin tubing, *C*, having a small opening at *F* (not necessary if two burners are used) is inserted in the large tube and the opening between the two stopped by a piece of rubber tubing, *D*. The tube, *C*, is attached by rubber tubing to the burner and the tube, *E*, to the gas supply. The joint, at *G*, is to allow the apparatus to be inserted in the opening of the air-bath. To regulate, run the temperature up until the thermometer reads the maximum desired, then push the tube, *C*, down until it just goes below the surface of the mercury. This "Thermostat" is very delicate, but the rubber ring, *D*, sticks to the tubes and makes it hard to move the inner tube, *C*, up and down.

## CHAPTER X.

### PREPARATION OF DISTILLED WATER.

**Automatic Stills.**—For the preparation of distilled water in the laboratories, nothing are quite so handy as the automatic laboratory stills sold by various dealers in chemical supplies. One of the best forms of these is the Jewell automatic water still, another good form is the Rochlitz automatic water still, Fig. 47, made for The Scientific Shop, 324 Dearborn Street, Chicago. This latter form the author has in his laboratory and it is probably necessary to operate it not more than a third of the time to supply all the water needed for the analytical work. It gives the

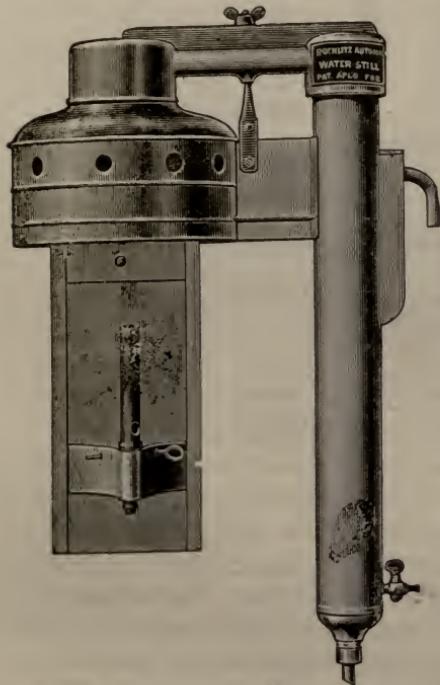


Fig. 47.—Rochlitz Automatic Water Still.

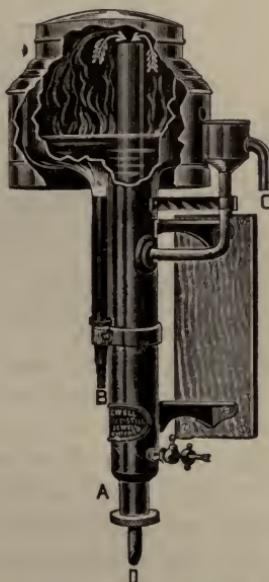


Fig. 48.—Jewell Automatic Water Still.

purest water of any of the small stills which we have examined. When pushed it will distill at the rate of about one gallon every two hours. Its price was \$15.00.

The Jewell still, Fig. 48, is made in a number of sizes, the smallest costing \$12.00 and having a capacity of  $\frac{1}{2}$  gallon per hour and the largest costing \$60.00 with a capacity of  $1\frac{1}{2}$  gallons an hour. These stills are all meant to be heated by gas, though the smaller sizes may be heated by a gasolene burner. The Rochlitz still may be heated over a small oil stove as it differs somewhat in construction from the Jewell still.

**Condensing Steam from Boilers, Etc.**—Other forms of automatic water stills are on the market and are described in various

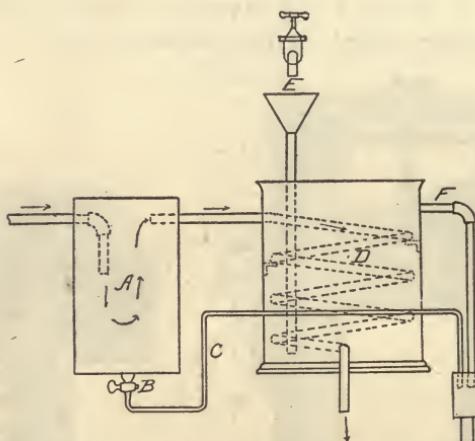


Fig. 49.—Condenser and Trap for Waste Steam.

dealers' catalogues. The automatic stills are so cheap that it hardly pays to get a copper retort and tin worm to distill water. While a few dollars first cost may be saved by purchasing these, the extra trouble of watching the latter, in order to prevent its boiling dry, and the extra amount of gas required to do the work, negative any such saving in a few weeks. Steam from the heating line may also be condensed for distilled water. In this event a trap should be interposed between the steam line and the block tin pipe coil in order to catch any drip from the iron pipe. Figure 49 shows an arrangement of this kind. *A* is the trap. It is a small drum made of galvanized iron, planished copper or iron pipe. It is provided with a drip cock, *B*, and a small siphon, *C*,

which will automatically draw off the water when it reaches a certain height. *D* is the worm. It should be made of block tin pipe. The cooling water enters at *E* and leaves at *F*. If the steam is drawn from the boilers of a large plant it is apt to contain oil, which will condense with the water and the latter will appear milky and smell of kerosene. While for some work this may not be objectionable, still such distilled water makes the beakers greasy and the burette levels hard to read, so that it is far better to get one of the automatic stills either heated by gas or steam. Where gasoline lamps are used the Rochlitz still, men-

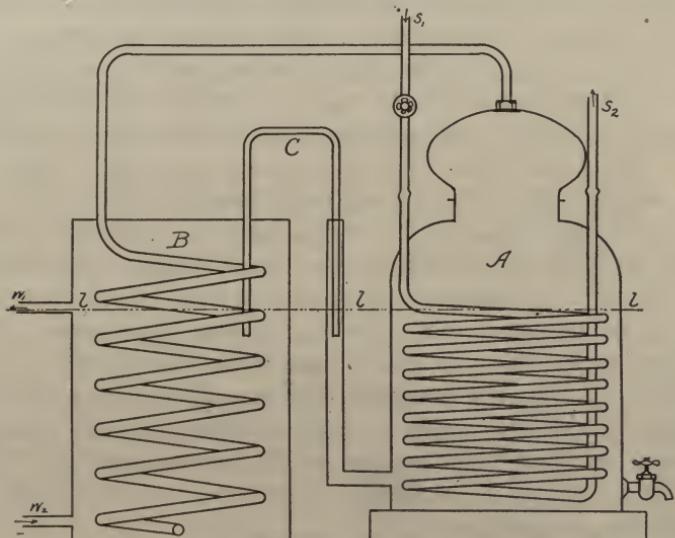


Fig. 50.—Still to be Heated by Steam.

tioned before, may be used and heated by a coal oil stove, if the gasoline lamps are troublesome or the gas supply is limited.

**Large Stills.**—When large volumes of water are needed a large size Jewell still heated by live steam from the boilers of the plant will be found most convenient and economical. They are made in sizes ranging in capacity from 3 to 30 gallons per hour and hence are large enough to meet any laboratory demand. The F. J. Stokes Machine Co., 17th and Cambria Sts., Philadelphia, also make automatic stills of great capacity and efficiency to be heated by live steam.

A retort heated by steam, such as is shown in Fig. 50, may

also be attached to the condenser illustrated in Fig. 49. This is made of copper and is heated by means of a coil of  $\frac{3}{4}$ -inch copper pipe coiled around the inside of the retort. This coil is connected with the steam line. The steam from the retort passes up into the dome and from this into a worm through a block tin pipe. As the water boils away from the retort more is automatically supplied through the siphon tube shown.

**Containers for Distilled Water.**—For holding distilled water one or two-gallon bottles are best, because one can be filling while the other is being used, etc. Distilled water attacks glass, dissolving silica, so that it should not be kept any great length of time. For this reason it is best to get a still which without pushing will distill a day's supply of water in a morning, and have freshly distilled water on hand every day, using one day the water prepared the morning before, and emptying each bottle before using the next. Tanks lined with block tin are used, as distilled water attacks tin very little. They should be soldered with pure tin. Carboys, stone jugs, jars, etc., of large capacities should not be used to hold distilled water in laboratories where inorganic gravimetric determinations are made, as water kept in such vessels is sure to become contaminated with silica, etc., giving high results. For the same reason the practice of using large stills heated by coal fires and run only now and then, long enough to fill up tanks or bottles with sufficient water to supply the laboratory for a month or two, is decidedly objectionable, on the score of accuracy. Distilled water should be occasionally tested for contaminations by evaporation in a weighed dish.

## CHAPTER XI.

### APPARATUS FOR ELECTROCHEMICAL ANALYSIS.<sup>1</sup>

Electrolytic determinations are now part of the routine of many commercial laboratories and means for carrying out such work will usually be found in every well-equipped metallurgical laboratory, no matter how small it may be. In determining copper, nickel, bismuth, etc., the electrolytic methods are far more satisfactory than the precipitation or volumetric ones, hence in a laboratory where ores containing copper are likely to be brought for analysis or where bearing metals or alloys, nickel, steel, etc., are analyzed means should be provided for electrolytic work.

**Use of the Electric Lighting Current.**—Stillwell and Austin<sup>2</sup> were, I believe, the first to suggest the use of the electric light current for the determination of metals in the electrolytic way, and the writer has found this to be not only sufficient for ordinary technical purposes, but also by far the most convenient. When available, it is much cheaper than batteries and saves the operator much trouble. The only disadvantage in connection with its use is the constant voltage which is usually 110 or 220. Indirect or alternating currents must of course be transformed to direct currents. This necessitates the use of a transformer, which may make the cost of installation of the outfit for electrolytic work so great as to make the use of batteries preferable to that of the electric light current.

It is of course necessary to reduce the strength of the current. For this purpose the resistance board shown in Fig. 51 and designed by the author is convenient and satisfactory. It consists of a stout board, 2 inches thick and 24 inches by 8 inches. This board should have its top covered by a piece of asbestos board, to guard against fire, etc., so that the apparatus may be left over night without danger. In a line down the middle of this board,

<sup>1</sup>Part of the material in this chapter is taken from articles by Dr. W. H. Easton in the Chemical Engineer, April and September, 1905.

<sup>2</sup>Jour. Anal. and App. Chem., VI., 129.

with their centres 3 inches apart, seven porcelain keyless sockets or receptacles,  $b_1, b_2, b_3$ , etc., should be fastened. The best form of receptacle to use is that shown in the cut. Single pole knife switches,  $a_1, a_2, a_3$ , etc., are also placed three on each side, as shown in Fig. 51. At either end of the board a binding post  $d^1$  and  $d^2$  is placed. Each receptacle is connected to the next receptacle and also to one point of the switch, by means of short pieces of insulated copper wire. The end receptacles are also connected, each to a binding post. All the connections should be made as shown.

With this board should also be provided six 16-candle power

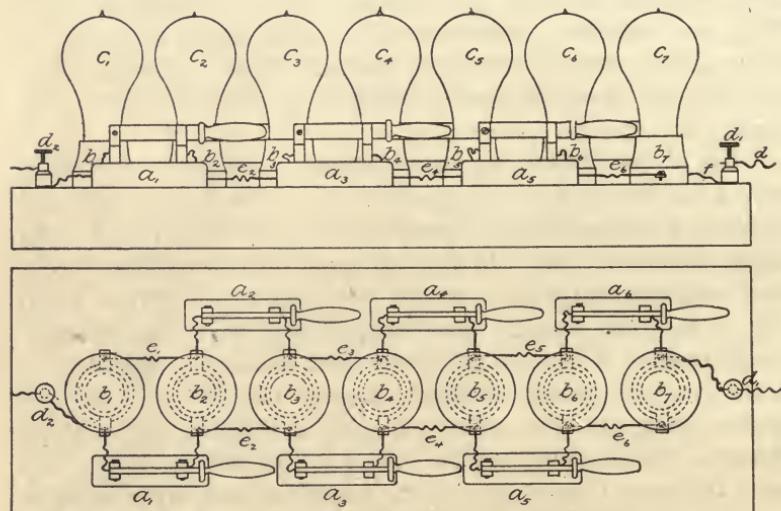


Fig. 51.—Lamp Resistance for Electrochemical Work.

lamps, six 32-candle power lamps, six 50-candle power lamps, and a safety plug of say 10 amperes. To use the board, connect one of the binding posts to one terminal of the electric light current and connect the other with one of the electrodes of the solution to be electrolyzed. Now, if a weak current is desired, screw 16-candle power lamps into the sockets and open all the switches. The current will then travel through all seven of the lamps, as follows: —from  $d_2$  through the lamp  $c_1$ , and from this to the lamp  $c_2$ , by means of the wire  $e_1$ , and thence to the binding post  $d_1$  through  $e_2, c_3, e_3, c_4, e_4, c_5, e_5, c_6, e_6, c_7$ , in order. For a resistance of six lamps, remove the lamp  $c_7$  and place the safety plug in the recep-

tacle  $b_7$ . For five lamps, close the switch  $a_6$ ; for four lamps, the switch  $a_5$ ; for three lamps,  $a_4$ ; for two lamps,  $a_3$  and for one lamp,  $a_2$ .

For stronger currents the lamps may be put in parallel. To do this, *take out the safety plug from  $b_7$* , close all the switches and put in from one to seven lamps, according to the strength current desired. Seven lamps will give about 3.5 amperes, while two lamps will give about one ampere. For yet stronger currents put in the 32-candle power or the 50-candle power lamps. For very weak currents, use another board and shunt the current through this around a light. For analytical work, however, the above board is sufficient and only 16-candle power lamps need be used, unless the rotating anode is to be employed, when 50-candle power lamps will be needed, since the currents for this work are much stronger.

The above board provided with 16-candle power and 50-candle power lamps will give a range of current of from about 0.07 ampere to about 11.0 amperes. The resistance of the board can be calculated, sufficiently near the truth for analytical purposes, by assuming for a 110 volt circuit, that the 16-candle power lamps have a resistance of 220 ohms; the 32-candle power lamps, 108 ohms; and the 50-candle power lamps, 69 ohms. If, therefore, seven 16 C. P. lamps are in series, according to Ohm's law:

$$\text{Amperes} = \frac{\text{volts}}{\text{ohms}} \text{ or Amperes} = \frac{110}{220 \times 7} = \frac{1}{14}.$$

Or the resistance of the lamps is  $\frac{1}{14}$  ampere or 0.07 ampere. If now the seven 16 C. P. lamps are in parallel the resistance will be  $\frac{110}{220} \times 7$  or 3.5 amperes.

Instead of calculating the resistance of the board, it is preferable to determine it by means of an ammeter. This may be done once for all, and the values for each combination of lights recorded in a note book, or the measuring instrument may be introduced into the circuit while the determinations are being made. This latter plan is preferable, but necessitates the possession of an ammeter by the laboratory, while in the former event, the work of determining the resistance may be done in some electric light station, or by some friendly electrician.

To start with a low resistance and increase to a greater, place all seven lamps in the board and open all the switches and connect up the apparatus with the terminal wires. Now take out the lamp  $c_7$  and screw the safety plug in the receptacle  $b_7$ . Then close in turn the switches  $a_6$ ,  $a_5$ ,  $a_4$ ,  $a_3$ , and  $a_2$ . *Do not close the switch  $a_1$* , however, or short circuiting will take place. Now *take out the plug* from the receptacle  $b_7$  and unscrew all the lamps, except  $c_1$ , from the receptacles, far enough to break the contact. Close the switch  $a_1$  and screw in the lamps to make contact one by one.

To avoid short circuiting, the connection between  $b_1$  and  $d_2$  or between  $b_7$  and  $d_1$  should be made with fuse wire, or another lamp receptacle may be put in between the receptacle  $b_7$  and the binding post  $d_1$ , connected to  $b_7$  and  $d_1$  by pieces of wire, and a safety plug kept in this all the time.

The board may be fastened to the wall, or a cord and plug socket may be fastened to it so that it can be put away in a desk when not in use. When wanted the board can be readily attached to the current by screwing the plug socket into a lamp receptacle, on the wall or hanging from the ceiling.

**Gravity Cells.**—When the electric lighting current is not at hand, batteries must, of course, be used. For ordinary analytical work six crowfoot or gravity cells will be found sufficient and these can be arranged in series or in parallel as the case may require. No resistance board is needed, as the strength of the current can be controlled by the number and the arrangement of the cells themselves. Where currents of greater strength than one or two amperes are needed the bichromate cell should be used in place of the gravity cells. When weak currents are to be used, however, nothing will be found so easy to take care of as the crowfoot or gravity cells. These may be arranged in a closet under the work-table on which the electrolytic work is done. They should be connected up to a board as shown in Fig. 52, so that they can be readily arranged in any way desired.

This board consists of two narrow strips of copper or brass,  $E$  and  $D$ , running along the front edge or on the side of the work-bench just under the top. These strips should have a binding post at either end of which the wires leading to the anode and cathode of the solution to be electrolyzed should be connected, and should be about 6 inches apart. Equidistant between the two

strips of metal twelve short pieces of brass spring  $a_1, b_1, a_2, b_2$ , etc., should be screwed into the board. These springs should be about  $3\frac{1}{2}$  inches long and should reach to either the strip  $D$  or  $E$ . Each pair of springs should be placed so that the adjacent spring of the following pair can rest on the screw holding the spring itself to the board. That is, referring to Fig. 52, the spring  $a_2$  should be so placed that it can be swung around so as to rest on either the copper strips  $E$  or  $D$  or on the screw holding the spring  $b_1$  to the board. Each spring should be connected to a pole of a

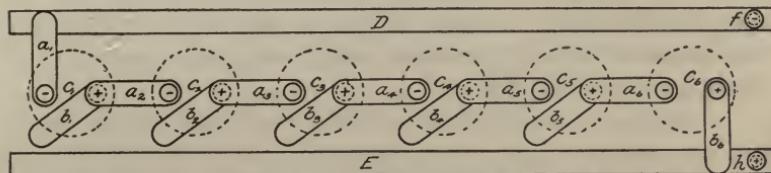


Fig. 52.—Board to Facilitate the Arrangement of Batteries.

battery. The springs  $a_1, a_2, a_3$ , etc., should be connected to the zincs of the cells, and the springs  $b_1, b_2, b_3$ , etc., should be connected to the coppers of the cells. Fig. 53 shows an elevation of one of the springs.  $A$  is a piece of brass spring with a round hole bored in one end and the other turned up to form a handle. This spring is fastened to the table by means of the brass screw  $B$ , and should be held in place by two washers, as shown.  $C$  is a piece of copper wire connecting the spring with the battery.  $D$  is the long copper strip running the length of the table.

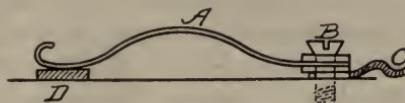


Fig. 53.—Spring Connector for Battery Board.

With the springs in the position shown in Fig. 52, the six cells are in series. The spring  $a_1$  connected with the zinc of cell  $c_1$  rests on the strip  $D$ , and the spring  $b_2$  connected with the copper element of cell  $c_6$  rests on the strip  $E$ , while the copper elements of the cells  $c_1, c_2, c_3, c_4$ , and  $c_5$  are connected to the zinc elements of the next cell by means of the springs  $a_2, a_3, a_4, a_5$  and  $a_6$ . The springs  $b_1, b_2, b_3, b_4$  and  $b_5$  are out of service and rest merely on the wood.

To connect in parallel, the springs marked  $a_1, a_2, a_3, a_4, a_5$

and  $a_6$  should rest on the strip  $D$  and those marked  $b_1, b_2, b_3, b_4, b_5$  and  $b_6$  should rest on the strip  $E$ .

A battery of six crowfoot cells can be bought for less than \$5.00.

**Storage Batteries.**—Storage batteries are a very convenient source of current when means for charging them is at hand and a board like that described above will be found very handy for connecting them up. Their relatively high voltage and amperage make them more suitable than any other form of battery, while their discharge is very steady and dependable. They give a voltage of about two. A higher voltage is obtained by adding more cells in series, (*i. e.*, the positive pole of one to the negative pole of the next, and so on).

Cells can be obtained for any amperage capacity, the amperage required to exhaust the cell in eight hours being the standard. A cell of two and a half amperes for eight hours discharge is large enough for ordinary use, although it is convenient to have cells of a larger capacity so as to allow longer intervals between charges.

Unfortunately, storage batteries cannot be used unless there is some means at hand for charging them, and moreover they are very delicate and need careful attention to prevent them from being ruined. Explicit directions accompany the batteries, and if means for charging them, such as the lighting circuit and a rheostat sufficient to reduce the current to the normal charging rate, are at hand, nothing better for electrolysis can be obtained.

A description of storage batteries, their use and care is beyond the range and scope of this paper; an excellent chapter on storage batteries, written by Prof. F. B. Crocker, will, however, be found in a little book, "Practical Lessons in Electricity," published by The American School of Correspondence at Armour Institute of Technology, Chicago, Ill., which anyone interested in the subject of electrochemistry will do well to procure.

**Rheostat.**—Where batteries or storage cells are used some means other than the lamp board previously described must be provided, since the resistance of this is far too great for such sources of current. Rheostats are manufactured for this purpose, made up with coils of resistance wire, and a movable arm that can be placed so as to allow the current to pass through as much of the resistance as desired. Such rheostats are best pur-

chased. They should be constructed so that they will cut down the current from one cell, from full strength to about .105 ampere, in from ten to twenty equal steps; and be able to withstand the full current from all the batteries at once.

A water rheostat makes a very cheap and simple means of controlling the current. A glass or earthenware jar of about three quarts capacity is filled with a very weak solution of sulphuric acid. Two arc lamp carbons are attached to the terminals of the current in series and dip into the solution. The farther the carbons are apart the greater the resistance. By fastening one on the edge of the jar and hanging the other on the edge with a clip so that it may be placed anywhere, an excellent rheostat is obtained. The resistance in the solution can be decreased by

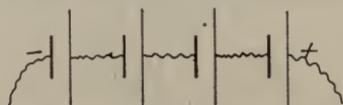


Fig. 54.—Diagram Showing Arrangement of Cells in Series Parallel.

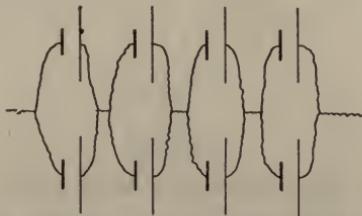


Fig. 55.—Diagram Showing Arrangement of Cells in Parallel.

adding acid, and increased by adding water. The size of the jar, etc., will depend upon the conditions of the circuit, and are easily found by experiment.

**Arrangement of Cells.**—The diagrams show how certain results can be obtained by altering the arrangements of the connections. It must be noted that a voltage of above the desired figure must be obtained by adding batteries in series and then cutting down to the right point by the rheostat. As mentioned before, by the series arrangement, the cells add their voltage to the circuit, but the amperage remains about the same as for one cell. Fig. 54 represents the series arrangement, in which the short thick lines represent the zins and the long thin lines the coppers. The irregular lines represent connecting wires. If  $3\frac{1}{3}$

volts are desired, the voltage is brought up to four by adding four cells, and then reducing the voltage to  $3\frac{1}{2}$  by the rheostat. This arrangement will give an amperage depending upon the resistance in the circuit and electrolyte. It should in most cases be sufficient, since the current usually required is very small, but if it is not, the amperage can be doubled by adding an equal number of cells in parallel. Fig. 55 represents this arrangement. This is a series parallel arrangement, giving the same voltage as in Fig. 54, but twice the amperage. It will be noticed that there are four groups of cells. In each group there are two cells with the zinc connected to zinc and the copper to copper. But the zincs of one group are connected to the coppers of the next, and so on. In making these arrangements, the same proportions always hold true, so that any combination of cells, to give almost any current, can be obtained.



Fig. 56.—Platinum Spiral Anode.

**Electrodes.**—In electrochemical analysis, a platinum wire or spiral is usually used as an anode, Fig. 56, and a platinum dish, cone or cylinder for the cathode. When the deposit to be weighed is formed on the anode the cone or dish is used as the anode, and the spiral for the cathode. Aluminum cathodes have been proposed for copper analysis, and even copper ones would probably give satisfactory results in this case. When obtainable, however, platinum should be used.

The most satisfactory cathode is a small platinum dish weighing about 30 to 50 grams. When large volumes of solutions have to be electrolyzed the cone or cylinder must of course be used and the solution held in a beaker.

**Stands.**—Figure 57 shows a convenient stand for use when a platinum dish is employed as a cathode. It consists of a wooden block, *A*, about 2 inches thick and 4 or 5 inches square, depend-

ing on the size of the dish. A round hole about an inch deep and about 3 inches in diameter should be gouged out of this and a triangle, *C*, of copper wire fastened above this, as shown in the illustration, by two round-headed screws *f*<sub>1</sub> and *f*<sub>2</sub>, and a binding post, *e*. A light wooden frame, *B*, should be fastened to the block, *A*, and the binding post, *d*, screwed midway in the top piece of this. The binding post, *d*, should be for two wires and the second

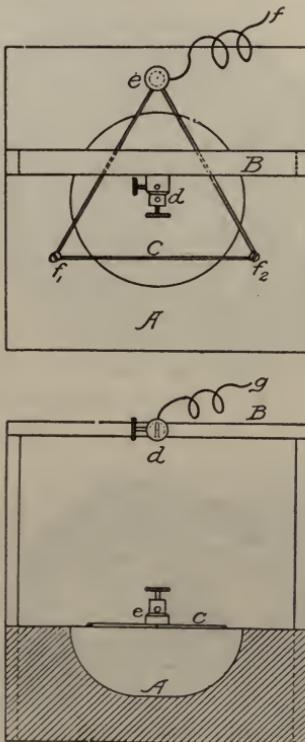


Fig. 57.—Compact and Simple Stand for Electrochemical Analysis.

hole should be directly over the centre of the triangle. The dish to be used as a cathode should rest on the wire triangle and the platinum wire to be used for an anode should pass through one hole of the binding post *d*. The wires carrying the current can then be attached one (+) at the vacant hole of the binding post *d* and the other (—) at the binding post *e*. When a cone or a cylinder is used as a cathode, the block, *A*, should be made solid, without the hole, and the frame should be high enough to permit

a beaker to be moved in and out under it comfortably. Another binding post similar to *d* should then be fastened beside *d* to hold the cone.

Very often supports similar to retort stands, except that the supporting rod must be of heavy glass to avoid short circuits, are

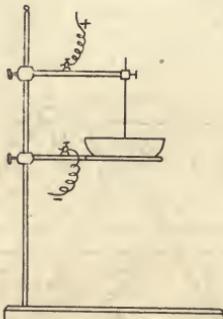


Fig. 58.—Support for Electrochemical Analysis.

used. Figure 58 shows such a stand. The supporting arms or rings can be obtained with binding posts to receive the terminals, and must be of bright metal, not painted. If the platinum dish is used it sets directly upon a ring clamped to the glass supporting



Fig. 59.—Ammeter—Weston.

rod. It is preferable to provide this ring with three platinum points for contact with the dish. If the dish is to be heated, a sheet of asbestos is fastened on the under side of the ring in order that heat may be applied to the solution without the dish coming in contact with the bare flame.

**Measuring Instruments.**—For measuring the current there will be needed an ammeter and a voltmeter.

The *Ammeter*, Fig. 59, measures the amperage or amount of current. The instrument should read from  $\frac{1}{100}$  of an ampere to at least two. It is connected with the circuit in series, as in Fig. 61.



Fig. 60.—Voltmeter—Weston.

The *Voltmeter*, Fig. 60, registers the volts or force of the current. It should read from  $\frac{1}{4}$  to 10 volts. It is placed as mentioned before in parallel, and between the precipitating vessel and all other parts of the circuit.

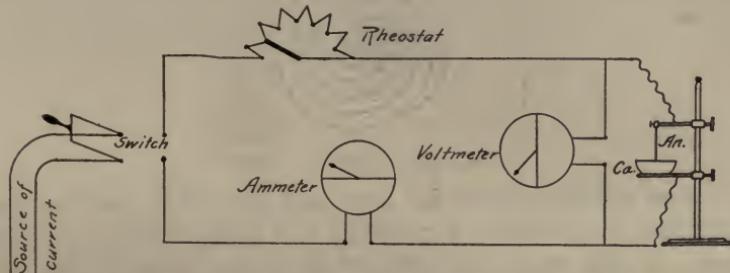


Fig. 61.—Assembling the Circuit Where Current is to be Measured.

Where the current is to be measured, the proper method of assembling the circuit is shown in Fig. 61. It will be noted that the rheostat and ammeter are in series and the voltmeter in parallel. There must be no other instrument between the voltmeter and the precipitating dish, as the true voltage between the anode and cathode will be given by this position only.

**Rotating Anode.**—Many electrochemical determinations can have the time necessary for complete deposition of the metal greatly shortened by rotating the anode. Means for doing this are described below. For the *cathode* a platinum dish is best suited.

The *anode* used is a spiral about two inches in diameter, made of heavy platinum wire. It is made slightly bowl-shaped so that it will remain immersed in the liquid in spite of the funnel-shaped form assumed by the latter on rotating the anode. Figure 62 shows its construction.



Fig. 62.—Rotating Anode.

Any suitable means for revolving the anode may be used providing the rotations per minute are high enough. A water motor, electric motor, or even power derived from shafting will answer. But undoubtedly an electric motor run by the lighting circuit is the most convenient. The anode is driven by a small round belt, and the use of cone or stepped pulleys will be found convenient, as several speeds can be gotten in this way. Figure 63 shows the general arrangement.

The anode should revolve at about 700 revolutions per minute.

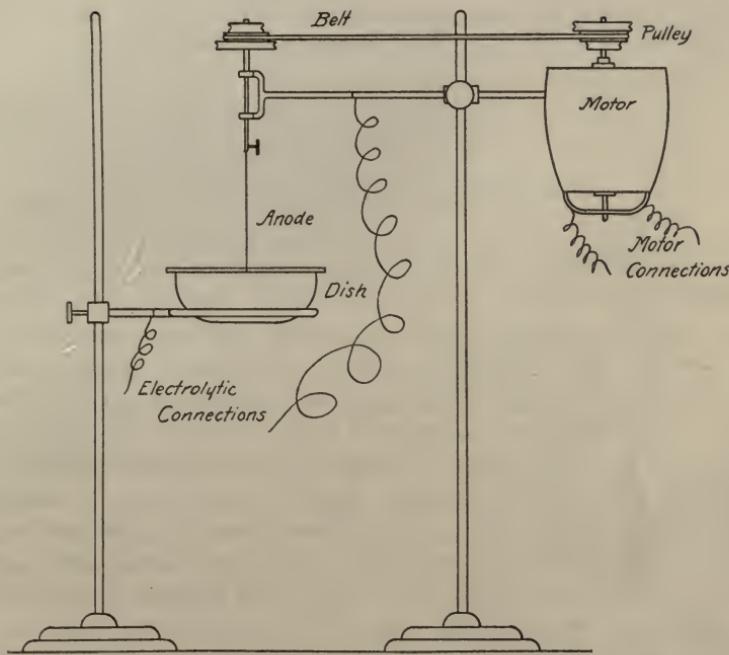


Fig. 63.—Apparatus for Electrochemical Analysis by Means of Rotating Anode.

When many determinations have to be made at the same time, one motor may of course be made to do for all by crossing the belt after each pulley. In this case the mortar and bearings of the anodes should all be mounted on the same framework.

## CHAPTER XII.

### SAMPLING APPLIANCES.

The apparatus used to reduce samples of ore, rock, coal, minerals, etc. from the dimensions usually encountered as they are brought to the laboratory, to the fine powder necessary for the analysis, may be divided into two classes, (1) crushers and (2) pulverizers. The former are used simply to prepare the material for the latter. Sometimes a pulverizer may be used also as a crusher. The ordinary iron mortar is an example of this, as it may be used not only to break down the large pieces of material to smaller sizes, but also to reduce these latter to a fine powder. Where small samples, of say from one to five pounds, alone are met with, the iron mortar will answer this purpose fairly well, particularly if much of this work does not have to be done, and in many small laboratories the small boy and the iron mortar will be found an excellent combination for preparing samples for analysis.

**Mortar and Pestle.**—Iron mortars can be obtained in a variety of shapes and sizes. Usually for this work, however, two mortars will be found handy, one of about a quart capacity and one holding about 2 gallons. These mortars should have their inside surfaces chilled. This will make the wearing surface of the mortar hard, while the iron itself will be tough enough to withstand the blows of the pestle. If the mortar and pestle are made of steel, they should be tempered, and the temper drawn from the outside of the mortar, leaving the latter tough.

In pulverizing very hard ores, the wear and tear on the pestle and mortar is often enough to contaminate the sample, to an appreciable degree, with splinters, etc., of iron. If the material to be powdered is neither magnetic nor contains magnetic substances, the iron entering the sample from the mortar and pestle may be removed by the use of a magnet. For pulverizing samples of spiegel or white iron, mortars of high carbon steel, properly tempered and toughened as mentioned above, should be used.

These can be procured from dealers in chemical supplies, and are also useful for pulverizing very hard ores.

Mortars having handles, or trunnions, projecting from each side to facilitate lifting and emptying are also for sale. Very heavy iron mortars may be attached to a block and tackle, so that the mortar can be lifted and emptied conveniently. The attachment can usually be most conveniently made by means of a wrought iron band or collar clamping around the upper part of the mortar and having a ring welded to it, through which the tackle may be run.

Mortars can best be set upon large blocks of wood while the pounding is going on. In order to keep the pieces of material from flying out from under the pestle and escaping from the mortar, the latter should be covered with a large piece of leather, having a hole cut through the middle for the pestle to pass through. Mortars can usually be cleaned by merely brushing out well with a stiff bristle paint brush. When material clings to the side, however, this can be removed by triturating a little clean dry sand in the mortar and then brushing this latter out.

**Reducing the Bulk of Samples.**—In preparing large samples for the laboratory, it is usual to crush the material by degrees, reducing the bulk of the sample after each crushing. For example, suppose a sample of twenty pounds of limestone is brought to the laboratory, consisting mostly of pieces of stone as large as a hen's egg and under. The whole sample is first crushed to a size of about one-half inch particles, and this is then reduced to one-fourth its bulk by "quartering," and the portion retained, about 5 pounds, is reduced to pieces the size of a pea and smaller. The sample of this size is again "quartered," and the remaining sample, about 20 ozs., is crushed so that it will pass a 20 mesh sieve and from this coarsely ground powder about 1 or 2 ozs. is selected and powdered so fine that it will all pass a sieve of 100 meshes to the linear inch. For limestone, this would be fine enough, but for refractory ores and silicates, it would be necessary to select enough of this material (passing the No. 100 sieve) for the analysis, and pulverize so fine that no grit could be detected on rubbing over the back of the hand or biting between the teeth.

The size to which the sample should be crushed before reducing its bulk is usually determined by the nature of the ma-

terial. When a sample of several pounds is received at the laboratory, if the material appears to be homogeneous to the eye, it will be sufficient to reduce it to a size passing a half inch screen. If the material is not of a homogeneous nature but is streaked with bands or shot with pebbles of various other materials the crushing should be much finer—unless the sample is a very large one, say 10 lbs. or more. Judgment is required in preparing samples, and the amount of work to be done can often be greatly abridged by a careful study of the material. The object in all cases is to have the small sample of one gram, or even less, weighed out on the balance pan, representative of the large one. Whether it is or not can usually be checked in a simple manner, by preparing two small samples from the original sample. That is when the first "quartering" is done, select two quarters and treat each as if it were a different sample. If the preparation of the sample has been properly done, analytical results upon the two will check. However, it is not usually with the laboratory part of the work of sampling that fault can be found, but rather with the field work. This can also be checked by taking two samples of the same pile or car of ore, coal, etc., or from the same deposit of limestone, clay, etc.

For determining the degree of crushing to be done, sieves are most convenient, the material being made to pass each sieve in turn. A convenient way is to make a sample of 5 lbs. or more all pass a one-half inch sieve. This sieve can be made of ordinary galvanized wire screening, having meshes  $\frac{1}{2}$  inch square, by tacking on to a wooden frame 18 inches square. The sieving may be done over paper or a sheet of light oil cloth. It can best be done, however, over a zinc or galvanized iron pan, 30 inches square and having a wall, 2 inches high, running around it, except for one corner, which is left open, in order that the pan may be conveniently emptied. The sample passing a  $\frac{1}{2}$  inch screen is then "quartered" and the quarter selected is made to all pass a screen having meshes  $\frac{1}{4}$  inch square, or 16 to the square inch. This sample is now quartered or, if still very large and the material is homogeneous enough to admit of it, its bulk is reduced to about one pound and all of this is made to pass a sieve having 20 meshes to the linear inch. This sieve can be bought, and is what is known as a No. 20 sieve. The sample passing this sieve can then be reduced to one-quarter or even one-eighth its bulk and made to pass

a sieve having 100 meshes to the linear inch (known as a No. 100 sieve). If further grinding is necessary, in order to allow the acids, etc., to act on the powdered material, the sample is spread out on a clean piece of paper or oil cloth, divided into a number of squares and as much of the material as will be needed for the analyses is taken by removing, from each square, a little of the material, on the point of a spatula. This material is then placed in an agate mortar and very finely pulverized.

The operation of reducing the bulk of a sample is usually known as "quartering" and is conducted in the following manner: The sample is poured upon a piece of clean, tough paper or oil cloth, or with large samples, on a clean floor, and mixed well. It is then heaped into a large pile and divided into four quarters by means of a thin sheet of metal, or, if the sample is small, a spatula is used. Two of these quarters (these diagonally opposite each other) are then scraped and brushed away and the two remaining ones are again mixed, divided into quarters and two of these rejected as before. The sample will now represent one-quarter its original bulk, and if the quartering is repeated again one-eighth its bulk, etc.

Mechanical appliances for quartering have also been devised. That shown in Fig. 64 is simple and easily constructed and works nicely. It consists of a cone or funnel, *A*, made of sheet metal, terminating in a short piece of galvanized iron or tin pipe, *B*. This opens into a wooden box, *C*. The top of the box, *C*, is loose so that the funnel and top may be removed and the box emptied when necessary. The pipe, *B*, is divided into four sections as shown in Fig. 64, two of which, *f* and *g*, empty into the box, *C*, while the other two, *h* and *i*, discharge into the pans, *E*<sub>1</sub> and *E*<sub>2</sub>, by means of the pipes, *D*<sub>1</sub> and *D*<sub>2</sub>. In using this sampler, the material to be quartered is poured into the funnel *A*, and dropping through the tube *B* is divided into four parts, two of which fall into the box and are rejected, while the other two go into the pans, *E*<sub>1</sub> and *E*<sub>2</sub>. The cone, *A*, should be kept fairly full of material and stirred all the time, so that a solid stream of particles is passing through the pipe.

**Crushers.**—Various mechanical contrivances have been devised by different parties to lessen the labor of crushing and pulverizing with the ordinary hand mortar. One of these is to fasten a stout spring to one of the overhead rafters in the ceiling of the

sampling room, and attach the pestle to the other end of this. It is claimed by those who use this device that the spring does not perceptibly add to the force required to strike a crushing blow, while it does greatly aid in lifting the pestle. Such a spring should be about 18 inches long, and have coils  $1\frac{1}{8}$  inch in diameter made of the best steel spring wire,  $\frac{1}{8}$  inch in diameter. This

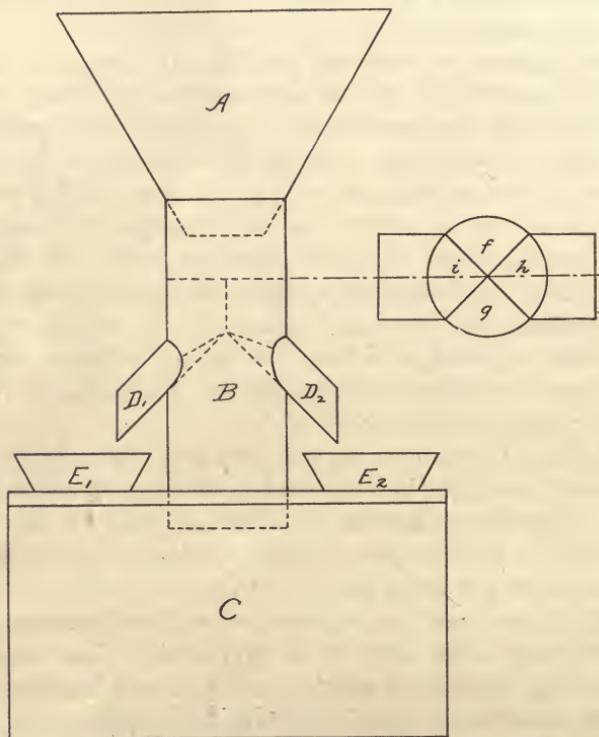


Fig 64.—Automatic Divider for Quartering Samples.

arrangement is of course only intended for use with a very heavy pestle.

Where power is at hand, the pestle may be raised by means of cams and allowed to fall of its own weight. Such an arrangement does not seem to have any advantage over the crushers, either in cost or effectiveness, and is nothing like so convenient. For crushing large lumps of ore, the chilled iron plate and pestle shown in Fig. 65 will be found useful. Its inside dimensions are  $24 \times 24 \times 3$  inches, and the walls and sides are 3 inches thick.

The operator stands, in using this plate, and merely lifts the pestle and allows it to do the crushing by its own weight.

Where large quantities of ore have to be broken up, small crushers will be found much more convenient than the mortar and pestle, and every laboratory which handles samples as large as ten pounds and over should be provided with some sort of mechanical crusher. If possible, this should be power driven, but

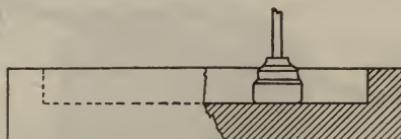


Fig. 65.—Chilled Iron Plate and Pestle for Crushing Samples.

even if it has to be operated by hand, it will be found more convenient than the mortar and pestle.

Figure 66 shows the Taylor Hand Crusher. This consists of two jaws, one of which is stationary and one of which is operated by the hand lever. Both jaws are faced with hard white iron. The movable jaw has horizontal corrugations, so as to force the material to be crushed down at each stroke of the lever. This jaw moves in both a vertical and horizontal direction. The lever

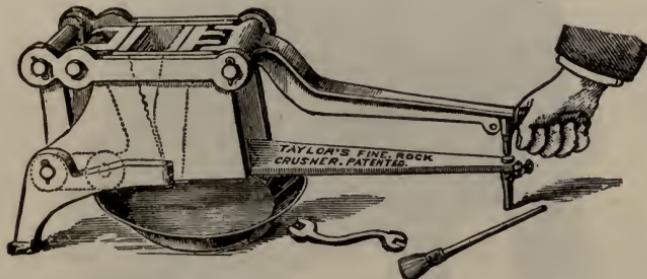


Fig. 66.—Taylor's Hand Crusher.

has a rubber covered hand grip, and a rubber cushion where it strikes the bed-piece, prevents jar and noise. The jaws are 3 inches wide and open  $1\frac{3}{4}$  inches, so that a piece of material  $3 \times 1\frac{3}{4}$  inches can be crushed. Forty pounds of the hardest rock can be easily crushed, in one hour, to such a fineness that 20 per cent. of it will pass a sieve having 60 meshes to the linear inch (No. 60-sieve). The jaws can be so adjusted that all can be made to pass the above sieve. The crusher can be easily cleaned and kept

in repair. It crushes much faster than a mortar, because the fine material drops out into the pan as fast as produced, and does not remain, as in a mortar, to deaden the blows of the pestle. This crusher is catalogued at \$15.00 and may be purchased of any dealer in chemical supplies.



Fig. 67.—Weatherhead's Crusher.

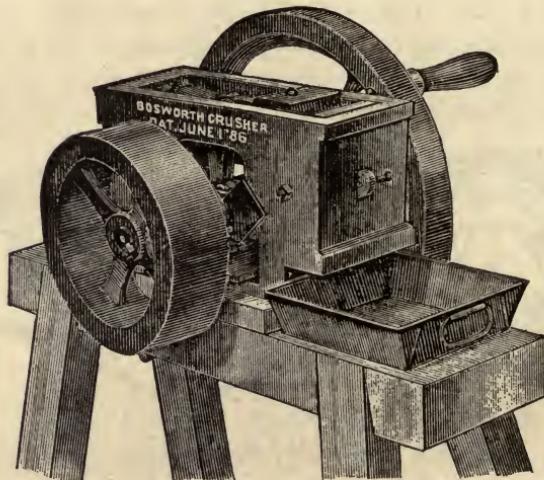


Fig. 68.—Bosworth's Crusher.

Figure 67 shows Weatherhead's Crusher, which is also a pulverizer. Its operation is evident from the cut.

Figure 68 shows a Bosworth Crusher, which may be operated either by hand or power. The Blake Crusher is somewhat similar to the Bosworth Crusher.

In these crushers one jaw is fixed and the other is swung

back and forth, through a very small arc, by means of an eccentric shaft, revolved by either the hand wheel or the pulley. The shaft in revolving raises and lowers a rod which is connected by toggles with the movable jaw. These crushers can be procured in sizes ranging from laboratory size up. The small size usually costs about \$50.00.

The Bosworth Crusher has been much improved upon of late, particularly with respect to the ease with which it can be adjusted and cleaned. Below will be found mention of three of these crushers.

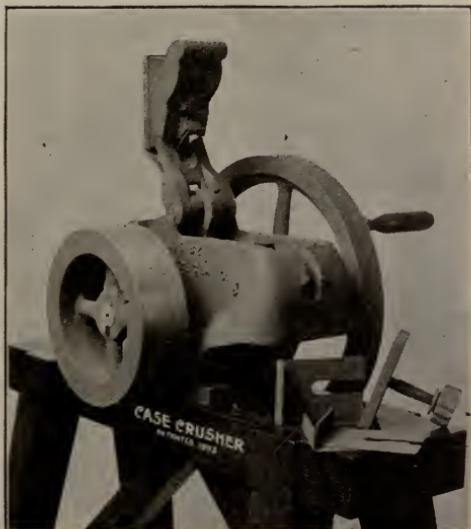


Fig. 69.—Case Laboratory Crusher.

The Case Laboratory Crusher (patented in 1903), Fig. 69, was especially designed to meet the demand for a low priced, strong, laboratory crusher. It can be driven by either hand or power, and will crush from 100 to 200 lbs. of ore per hour, depending, of course, on the hardness of the ore. The jaw opening is  $2\frac{1}{2}$  by 3 inches and it can be adjusted quickly from a fineness of  $\frac{1}{4}$  inch to 20 mesh. The feed is regular, and it is not inclined to cake on soft material. Messrs. E. H. Sargent & Co., Chicago, handle this crusher which they sell at \$30.00, hand driven, and \$32.00, fixed for power.

Figure 70 shows the Calkins Advance Ore Crusher, manu-

factured by The Calkins Company, Denver, Colo. One of the features of this crusher is the ease with which it can be cleaned. This is done by swinging up the front jaw of the crusher exposing the sides and face of the vibratory jaw and giving access to all parts of the machine to which material being crushed may adhere. The adjustment to coarse or fine crushing is made at the front end of the crusher. This machine is made in two sizes; the smallest has a jaw opening  $2\frac{1}{2} \times 3$  inches, weighs 170 pounds, and is listed at \$30.00 net; the larger size has a jaw opening  $3 \times 4$  inches, weighs 280 pounds and is listed at \$60.00 net.

The "Lightning" Crusher, Fig. 71, manufactured by F. W. Baun & Co., Los Angeles, Cal., is also a very efficient crusher

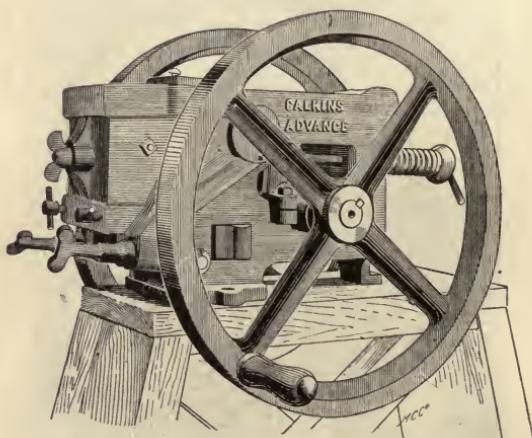


Fig. 70.—Calkins' Advance Ore Crusher.

which may be adjusted to crush fine or coarse. This crusher is also very easily cleaned, as, after lifting a pin, the front jaw may be swung out on a vertical hinge exposing both crushing plates and allowing them to be quickly and thoroughly cleaned with a brush. This crusher is very compactly and strongly built.

Where very large quantities of ore have to be crushed a Gates gyratory crusher will be found most useful. These are made by the Allis-Chalmers Co., Milwaukee, Wis., in a variety of sizes and types. The smallest size will crush 500 lbs. of material of average hardness per hour, to a fineness of  $\frac{3}{4}$  inch or 250 lbs. to  $\frac{1}{8}$  inch.

**Agate Mortar.**—For finely pulverizing small samples, already reduced by means of the iron mortar and pestle to pass a No. 100 sieve, the agate mortar is indispensable; except when the ore or mineral is very soft, when a Wedgwood mortar and pestle may be used. From the size and shape of the ordinary agate mortar and pestle the operation of grinding is very tedious. It may be greatly facilitated, however, by cutting a hole, of such size and shape as to hold the mortar firmly, in the middle of a block of hard wood, a foot or so square. The pestle is then fixed in a piece of round brass tubing of sufficient bore to hold it firmly, or else in a round hard wood handle.

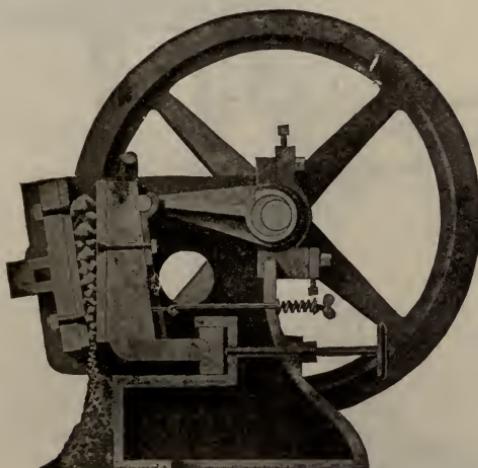


Fig. 71.—Braun's Lightning Crusher.

**Mechanically Operated Agate Mortar.**—Some ten or fifteen years ago Mr. Maunsel White, of the Bethlehem Iron Company, designed a mechanically operated agate mortar and pestle for use in the laboratory of the above concern. This grinder is illustrated and described in Blair's "The Chemical Analysis of Iron."

The McKenna Bros. Brass Co., Pittsburg, Pa., have also brought out a mechanically operated agate mortar and pestle. This is illustrated in Fig. 72. It consists of a revolving table on which the agate mortar is clamped, while the agate pestle is firmly fixed in a shaft revolving at a slight angle from the vertical. This machine reproduces almost exactly the motion used in hand grinding. The spring at the top of the sliding rod, to which the agate

pestle is fixed at the bottom, can be adjusted to give any desired pressure, or can be thrown back entirely to allow the pestle to be raised in removing the agate mortar. The mortar is readily removed by loosening a set screw and dropping one of the four posts holding the mortar in place. A scraper keeps the ore in the center



Fig. 72.—McKenna Mechanically Operated Agate Mortar and Pestle.

of the mortar, and the combined rolling and sliding motion of the pestle, which is controlled by a ball and socket side arm, reduces the hardest ore very rapidly, no attention being required from the operator. The grinder may be operated by any convenient power, of which not to exceed  $\frac{1}{8}$  horse-power is required. The mortar

used has a diameter of about  $4\frac{1}{4}$  inches. These mortars were introduced into many laboratories and gave good satisfaction.

The agate mortar is, of course, intended only for grinding very small quantities (5 or 10 grams) of ore at a time. It has one great advantage over other forms of pulverizers in that it can be used to reduce the hardest ores to a fine powder without danger of contamination of the sample from the wear of the mortar and pestle. When soft materials, such as coal have to be ground, large Wedgwood mortars and pestles are very useful. In using these, however, the material should be merely rubbed between the

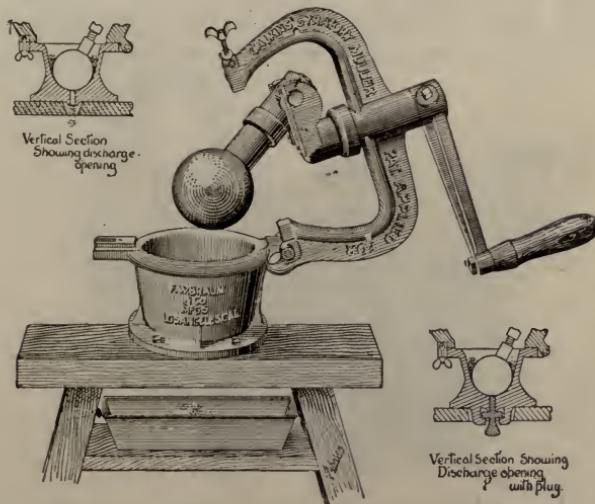


Fig. 73.—Braun's Gyratory Muller.

pestle and the mortar, and never pounded, for fear of contaminating the sample with splinters of porcelain.

**Gyratory Muller.**—Two excellent pulverizers, one of which can be operated by hand, are manufactured by Messrs. F. W. Braun & Co., Los Angeles, Cal. They are the gyratory muller and the disc pulverizer. The first of these is driven by hand and the second is intended to be power-driven from a small electric motor or the mill shafting.

The gyratory muller is shown in Fig. 73. In this muller the grinding is done by a ball or spherical pestle which revolves in a semi-spherical mortar with a particular twisting motion. This motion is imparted to the pestle by means of a right angle clutch

extension. The material is rapidly crushed by the revolving ball, and the twisting motion prevents the material from being thrown ahead of the ball, by centrifugal force, and causes it to discharge through an opening, provided for that purpose, in the bottom of the mortar.

A coil spring around the ball shaft gives tight engagement between the surface of the ball and the mortar, and more or less compression may be obtained by means of washers above the spring. Loosening the thumbscrew allows the base or arch to be swung back, and this lifts the ball from the mortar, giving access to the interior for cleaning. This machine has been found entirely satisfactory where large or small quantities of material are to be pulverized to a given size. The best results are obtained by

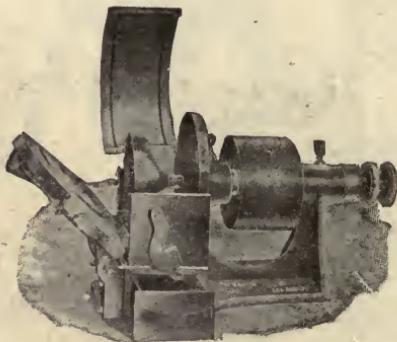


Fig. 74.—Braun's Disc Pulverizer.

screening the product each time, for if this is not done the finer particles surround the larger, preventing them from being pulverized. This appliance differs from the disc pulverizer described below, as it is necessary to feed and re-feed several times, according to the material, to obtain an extremely fine powder. This muller is priced at \$35.00.

**Disc Pulverizers.**—Messrs. F. W. Braun & Co. have recently brought out another form of fine grinder or pulverizer, for which they claim great efficiency and which they call a Disc Pulverizer, shown in Fig. 74. In this machine the grinding is done between a stationary and a revolving disc. The stationary disc or plate is fastened to the door of the machine. The meshing of these discs can be adjusted so as to regulate the fineness to which the material is to be ground.

This machine will pulverize any material that can be reduced to pulp on the old style bucking board. It will, with one feeding, pulverize an entire ore sample to any desired degree of fineness up to 200 mesh powder. An 8-ounce sample of ordinary granite rock can be reduced to 100 mesh in one-half minute. Other degrees of fineness may be obtained in proportionately more or less time. It may be thoroughly and quickly cleaned after each sample has been fed. The adjustment may be altered for the desired powder in a second. It is dust proof. There is no loss of material. Its pulverizing discs wear to place. The wearable parts, which have a very long life, are renewable and easily replaced. The discs are made of hardened steel with faces ground true. The bearings of

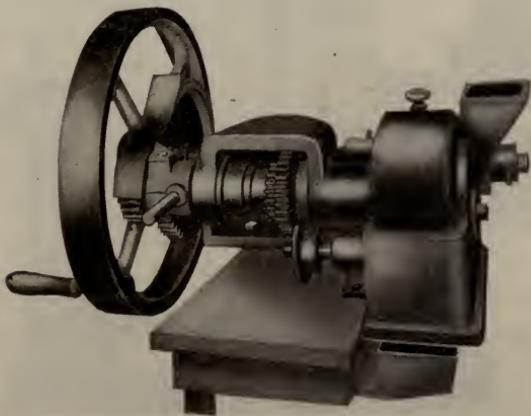


Fig. 75.—Calkins' Advance Disc Sample Grinder.

the revolving disc are babbitted and extra long. Provision is made for preventing the oil from entering the pulverizing chamber. The revolving disc is tapered from its face to the shaft, and is also machine finished to prevent fanning of material when in operation, and to economize power. At the bottom of the four sides of the pulverizing chamber, lips are provided, which ensure the discharge of material into the pan which fits closely into a slide beneath the discs. This machine is fed through the spout in the door, and will take ore  $\frac{1}{4}$  mesh and smaller, and reduce it all at one feeding to any desired mesh. Price of the machine, complete, \$85.00.

The Calkins Advance Disc Sample Grinder is shown in Fig. 75. It consists of a main frame or support to which is secured a

stationary disc. A rocker-arm, carrying a driven shaft, to one end of which is secured a grinding disc, and having driving gears and fly wheel at the opposite end, is pivotally journaled to the main frame of the machine. When in operation the disc on the driven shaft is given a double motion, rotating and vibrating. The oscillating or vibrating motion is created by means of an eccentric on the driven shaft which by rotating against a roller which is journalled to the main frame of the machine, causes the rocker arm carrying the driven disc shaft to rise and fall.

The cam or eccentric is not keyed to the disc shaft but is slowly rotated around the disc shaft by means of gears so as not

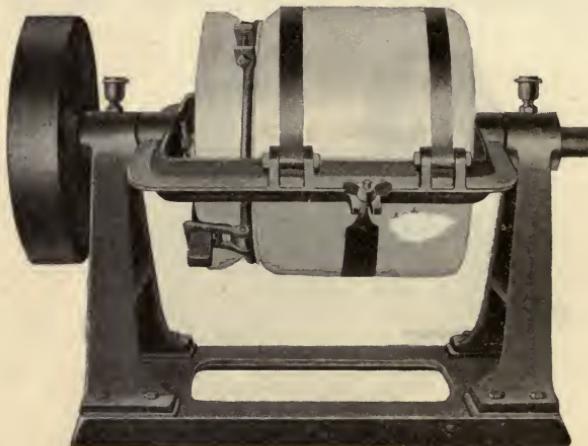


Fig. 76.—Abbé Jar Mill.

to limit the oscillating motion of the rocker arm and disc to a contracted orbit of travel. This double motion is very destructive to the ore particles which are fed between the discs from a hopper leading to an opening through the stationary disc, and not only alters the position of the rolling particles between the discs, but prevents the discs from becoming concentrically grooved, which would occur should the disc or discs have a rotary motion only.

The rocker arm carrying the driven shaft and disc may be lifted or swung over, clear of the opposing disc, to allow both discs to be cleaned of any adhering particles of any sample previously crushed, which might otherwise "salt" the succeeding sample.

Adjustment for coarse or fine grinding is made by means of lock nuts on the pivot shaft, which secures the rocker arm to the

frame of the machine. The lock nuts bear against the part of the frame through which the end of the pivot shaft passes, and retain the rocker arm and rotary disc in any desired degree of adjustment relative to the stationary disc. The price of the pul- verizer is \$50.00 and the weight 175 pounds.

**Jar Mill.**—The Abbé Engineering Co., 220 Broadway, New York, manufacture a small jar mill which is shown in Fig. 76. This mill consists of a porcelain jar,  $13 \times 12\frac{1}{2}$  inches, held securely in a cast iron frame by brass bands. Two of these bands are fastened at each end, but the third is held in place by a thumb-nut, which allows the jar to be taken from the frame. The frame is provided with a shaft at either end, and is revolved by a pulley as shown. The jar is closed by a porcelain cover, which is clamped tightly on the top of the former, a tight joint being secured by means of a rubber washer. The jar is half filled with porcelain balls. As the jar is concentric with the axle of the frame, the grinding is done by the pounding of the pebbles as the jar revolves. The speed of the latter is from 40 to 50 revolutions per minute, about 23 lbs. of porcelain balls are used for a charge and about 15 lbs. of material can be ground at a time. The mill is intended to be power driven, and may be run by a small motor.

These mills may be used for a great variety of laboratory work, and are very useful where large samples have to be very finely pulverized, as it is possible to grind extremely fine with them. Owing to the difficulty of cleaning the pebbles they are more used for experimental work than for preparing analytical samples. The pebbles may be separated from the ground material by using a large coarse mesh sieve, and they may be cleaned either by brushing with a stiff brush, or by washing with water or acid, or by grinding and discarding a little of the material to be pulverized.

This size jar mill is priced at \$60.00 and weighs 175 lbs. The Abbé Engineering Co. make a number of mills of this type, ranging in capacity from the size mentioned up to one of several tons capacity per hour.

## CHAPTER XIII.

### ASSAY FURNACES AND ACCESSORIES.

Assay furnaces are made to use three kinds of fuel—gaseous, liquid and solid. Those using gas are very little employed, usually only in city laboratories where but a few assays are made. They are, of course, very convenient when occasional assays are made,

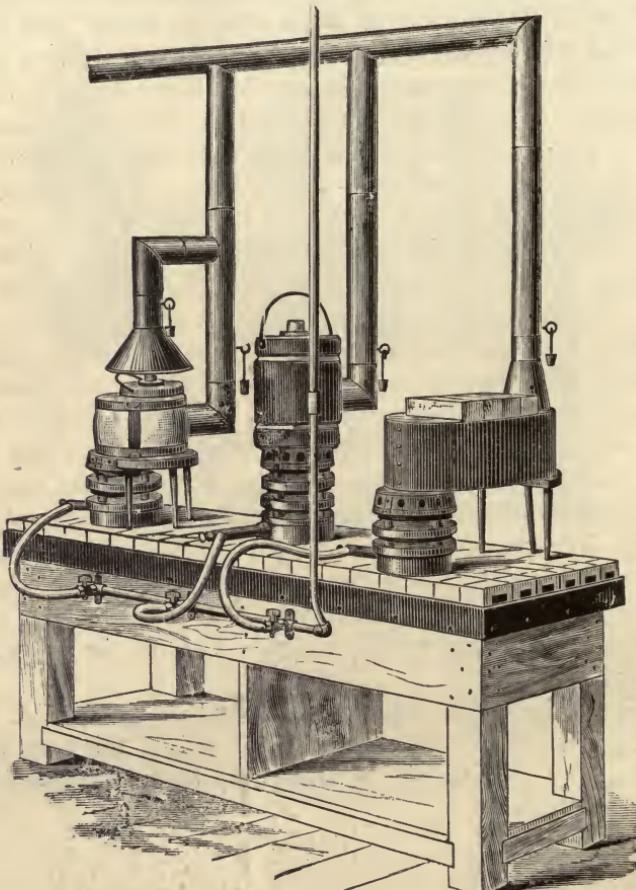


Fig. 77.—Assay Furnaces and Table—Buffalo Dental Mfg. Co.

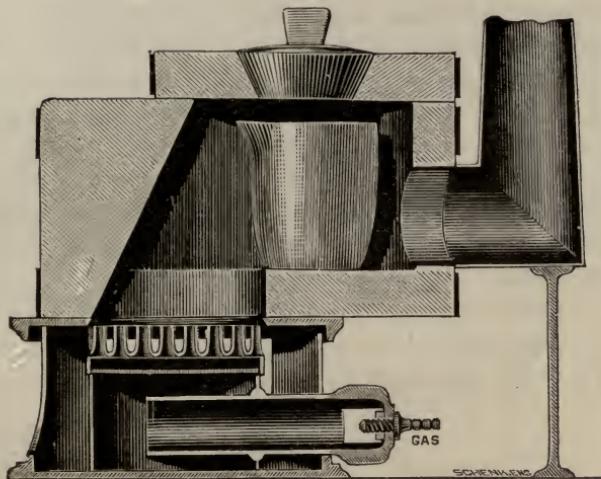


Fig. 78.—Roasting Furnace—Buffalo Dental Mfg. Co.

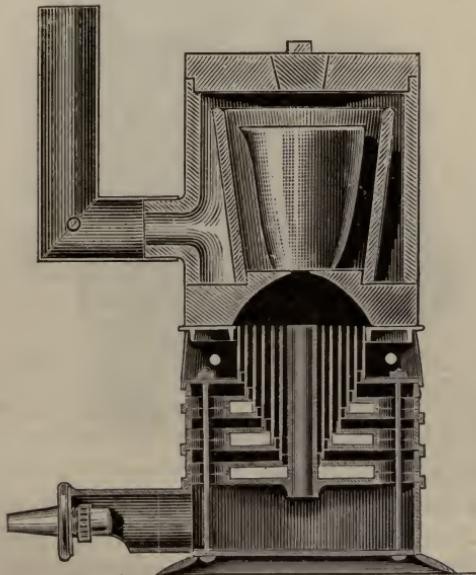


Fig. 79.—Crucible Furnace—Buffalo Dental Mfg. Co.

as in the laboratory of a consulting chemist, who is sometimes called upon to determine the value of a gold or silver ore.

Perhaps the best of these gas heated furnaces are those manufactured by the Buffalo Dental Manufacturing Co., Buffalo, N. Y. For assaying, three of these furnaces will be needed (see Fig. 77).

One for roasting sulphide ores, one for crucible fusions and one for cupellation. Figure 78 shows the roasting furnace, which is known as "No. 63, Direct Draft Crucible Furnace." It consists of a fire clay body strapped with sheet-iron bands, and provided with a Fletcher burner. The opening at the top, which is protected when not in use by a cover, is to allow the heat to have full play upon the roasting dish placed upon it. The hot flame passes through the furnace and up the chimney. When set up this furnace should have a hood or funnel placed over it as shown in Fig. 77. This carries off the odors and gases given off by the ores. The pipe and hood should be provided with dampers. This furnace is listed at \$12.00.

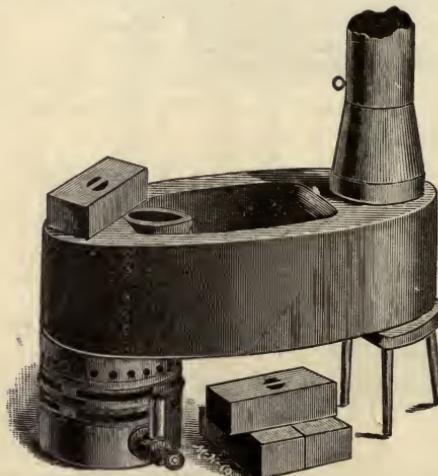


Fig. 80.—Cupellation Furnace—Buffalo Dental Mfg. Co.

The crucible furnace is shown in Fig. 79. The furnace is heated by a No. 15 Fletcher burner and is made in five parts—an outer cylinder of fine clay, which is covered by a removable fire clay top with handle attached, and rests upon a bed plate, also made of fire clay, which has a hole in it through which the flame passes, a graphite inner cylinder and a combustion chamber below. This furnace is listed as "No. 15 Crucible Furnace" and priced at \$16.00.

The cupellation and scorification furnace is shown in Fig. 80. This furnace was designed by Mr. Walter Lee Browne, and is described by him in his "Manual of Assaying." In form it is almost that of the reverberatory furnace, the movable bricks when

in place being the roof. In the interior, upon the bottom, are four little wedge-shaped bridges of fire clay, which are movable, and upon them rests a false bottom or floor, also movable. The latter corresponds to the muffle bottom of an ordinary furnace and upon it is done all the work. The furnace is heated by a No. 16 burner, and is made by the Buffalo Dental Manufacturing Co. It is called by them "No. 630 Monitor Furnace" and is listed at \$18.00.

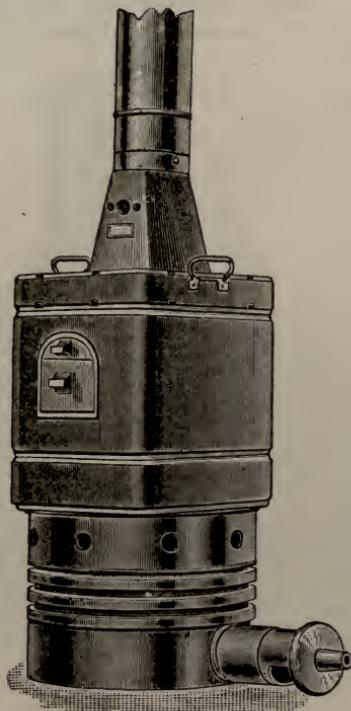


Fig. 81.—Muffle Furnace—Buffalo Dental Mfg. Co.

The table is of pine and is covered with fire clay tile. The whole outfit, table and furnace, is listed at \$75.00.

Another small gas-fired assay furnace, also manufactured by the Buffalo Dental Manufacturing Co., is shown in Fig. 81. It is made in four sizes, the smallest priced at \$17.00, and having an inside muffle space  $3 \times 4 \times 2\frac{3}{8}$ , and the largest costing \$45.00 and having a muffle space of  $6 \times 8\frac{1}{2} \times 4\frac{7}{8}$ . A simple outfit for assaying would consist of the No. 4 size of this, having a muffle space  $3\frac{1}{8} \times 5\frac{1}{8}$  and priced at \$20.00, for the cupelling and the

crucible furnace referred to before, which could be made to do the roasting also if set up with a hood over it. Some assayers, however, do not roast their ores.

**Liquid Fuel Furnaces.**—A large number of very convenient assay furnaces designed for the use of gasoline are now on the market, and where much assaying is done, and the assayer is not permanently located and gasoline can be obtained, nothing quite takes their place. They are small and can be quickly heated. They can also be easily transported from place to place. They

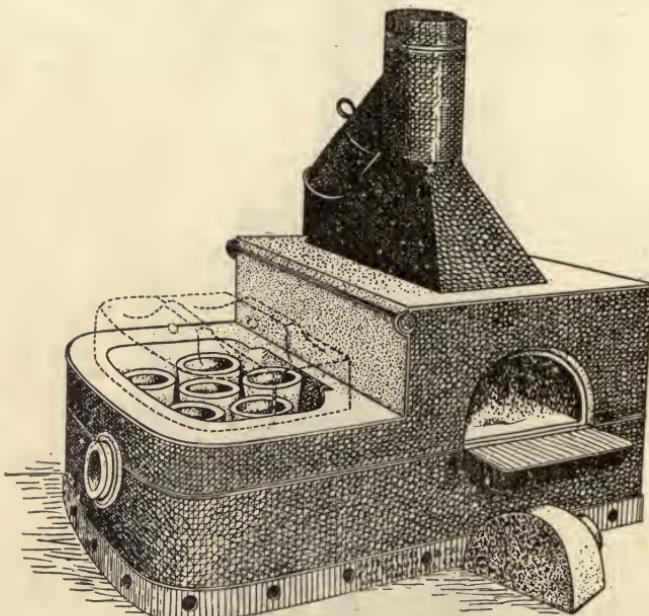


Fig. 82.—L. & C. Combination Furnace—F. W. Braun & Co.

are designed for either crucible fusions or for cupellations or for both. The latter, so called "combination furnaces," are the most convenient and economical. In them the fusion is carried on in one part of the furnace while the cupellation is being done in the muffle.

Fig. 82 shows the L. & C. combination furnace, manufactured by the F. W. Braun Co., Los Angeles, Cal. This furnace consists of a compartment for crucibles at one end and the muffle at the other. It has a burner hole at either end and is mounted on a swivel which allows the furnace to be revolved so that the

burner may be inserted in either one. Usually the burner is first inserted in the crucible end long enough to make one melt; the furnace is then revolved and the burner inserted in the muffle end. From this time on the melting and cupellation may be carried on at the same time without revolving the furnace. A dividing brick is furnished, to be placed between the melting and muffle departments, if only one is to be heated. A small trap-door is placed in the bottom of the crucible compartment, and if a spill occurs this may be opened, the bottom punched out and a new one of fire clay and sawdust put in. Oxidation in the muffle is secured by means of a fire brick flue connecting the muffle, through a hole in the latter, with the main flue by means of a pipe at the back

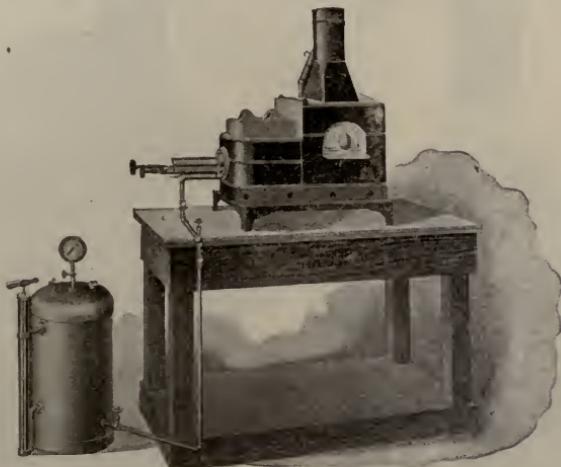


Fig. 83.—Cary Combination Furnace and Burner—F. W. Braun & Co.

of the furnace, as shown. The pipe is provided with a damper to regulate the draft when the buttons in the cupel are opening.

These furnaces are intended to be heated by the "Sunset" burner, made by the same firm. The arrangement of furnace and burner is illustrated in Fig. 83. The furnace itself should be raised slightly from the table, as shown, and the latter should be covered with asbestos, or better still, with fire tiles. The concrete table top mentioned in Chapter II. is suited to this, particularly if coarse calcined magnesia or asbestos fibre is used in place of sand, in the proportions of about three parts to one of cement.

This furnace is made in a number of sizes ranging from a  $4\frac{3}{4} \times 8 \times 3$ -inch muffle, holding two No. G crucibles to one having

a muffle  $6 \times 9 \times 4$  inch and taking four No. G crucibles. The price of the latter size together with burner, gasolene tank, pump, etc., is \$44.50. A furnace made similar to the one described above called the "Cary Combination Furnace," is to be heated by the "Cary" burner, a more efficient burner than the "Sunset." The price of the complete Cary outfit as shown in Fig. 83 is \$55.00, exclusive of the table.

The "Advance Combination Melting and Muffle Furnace No. 10," manufactured by The Calkins Company, is shown in

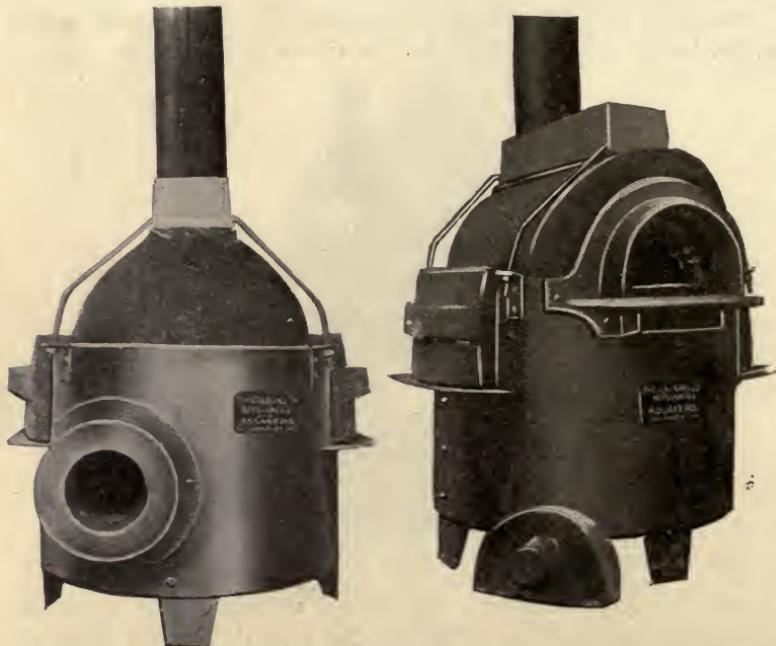


Fig. 84.—Advance Combination Furnace—The Calkins Co.

Figs. 84 and 85. This furnace consists of a circular crucible chamber above which is the muffle, Fig. 84. Access to the crucible chamber is had from either side of the furnace through covered apertures. The flame is shot into the crucible chamber at a tangent to the inner wall of the furnace, Fig. 85, and swirls around the crucibles. It is claimed that by not impinging the flame on the crucibles the life of the latter is thereby greatly increased and that the products of combustion rising unobstructed around the muffle, the latter is uniformly heated. The muffle is ventilated,

as shown in Fig. 84. The furnace itself is encased in an iron jacket and is provided with a removable bottom to allow replacement of the floor of the crucible chamber. The furnace is 16 inches in diameter and 21 inches high and weighs 180 pounds. The muffle is  $4 \times 6 \times 12$  inches and the crucible chamber is 12

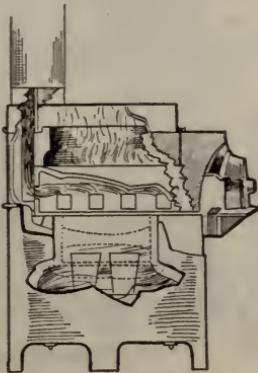


Fig. 85.—Advance Combination Furnace—The Calkins Co.

inches inside diameter and 8 inches high. Starting with a cold furnace, a good working heat can be obtained in 20 minutes and the muffle will be ready for cupelling before the melt is completed. The furnace is heated by an "Advance" hydrocarbon burner. The price of the furnace above is \$25.00 or of furnace, burner,  $7\frac{1}{2}$ -gallon gasoline tank and pump, is \$48.00. The "Advance" fur-



Fig. 86.

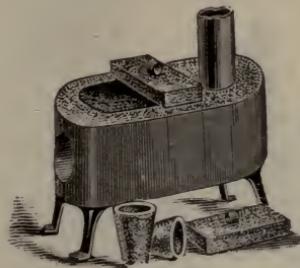


Fig. 87.

Crucible Furnaces—The Hoskins Co.

nace is made in two smaller sizes, one of which costs \$20.00 and the other \$16.00.

The Hoskins Company, 93 Erie St., Chicago, Ill., make several forms of gasoline-heated assay furnaces. Figures 86 and 87 illustrate crucible furnaces of which the first is intended for only one crucible, the second is made in two sizes taking respectively

two and four crucibles. Fig. 88 shows the muffle furnace. The price of the crucible furnace for one crucible, muffle furnace, and one gallon blow pipe outfit, complete, is \$40.00. Their combination furnaces are shown in Fig. 89 and Fig. 90. The first furnace shown is very light and portable and is intended for prospectors. It is also of use when only an occasional assay is made. On the right of Fig. 89 the furnace is shown prepared for crucible

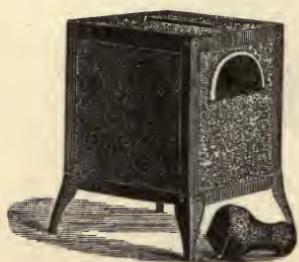


Fig. 88.—Muffle Furnace—  
The Hoskins Co.

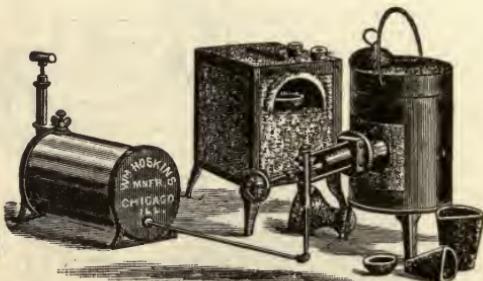


Fig. 89.—Prospectors' Combination  
Furnace—The Hoskins Co.

work. By lifting off the cover and substituting the part with the muffle opening, and sliding into this the muffle, the furnace is prepared for cupelling or scorification. The muffle is  $6 \times 3\frac{1}{2} \times 2\frac{1}{2}$  and the crucible furnace is 4 inches in diameter and  $5\frac{1}{2}$  inches deep. Its price with  $\frac{1}{2}$ -gallon blow pipe outfit is \$30.00. The combination furnace, shown in Fig. 90, is made in two sizes, the

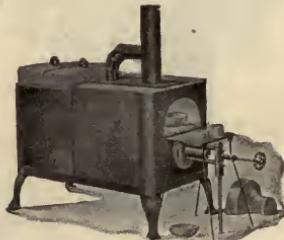
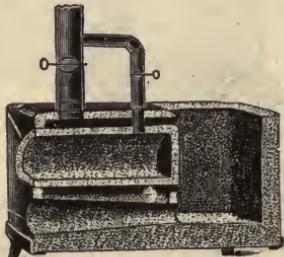


Fig. 90.—Combination Furnace—The Hoskins Co.

smaller of which has a muffle  $6 \times 3\frac{1}{2} \times 2\frac{1}{2}$  inches and a crucible compartment large enough for one fusion. The larger size is intended for four No. F crucibles and has a muffle  $10 \times 6 \times 4$  inches. The price of the smaller furnace is, with  $\frac{1}{2}$ -gallon blow pipe outfit, \$33.00, and of the larger one with a gallon blow pipe outfit, is \$46.00.

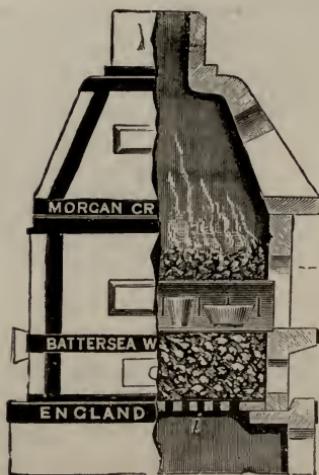


Fig. 91.—Battersea Assay Furnace.

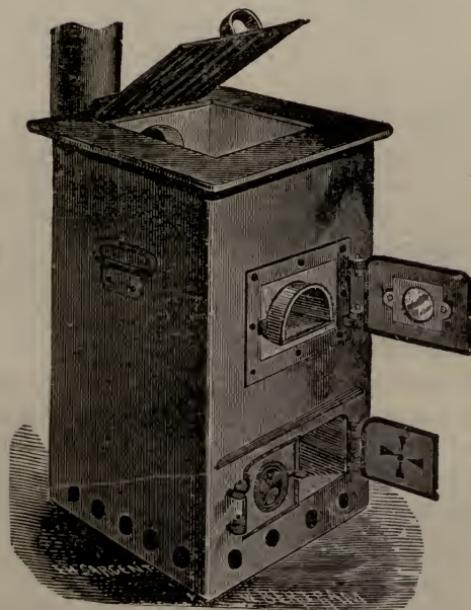


Fig. 92.—Brown's Portable Assay Furnace.

**Portable Coal-Fired Furnaces.**—Of the portable coal-fired furnaces, Bosworth's, the Battersea and Brown's forms are all extensively used in this country. The Battersea and Bosworth's furnaces are very similar and are made of fire clay, in sections, bound together with iron bands. They are muffle furnaces exclusively, but they are made in large sizes and in these latter crucible melts can be made in the muffle. These two furnaces cost about the same, the price ranging between \$30.00 and \$40.00 for the various sizes. The Battersea is shown in Fig. 91. Brown's Portable Assay Furnace is illustrated in Fig. 92. It consists of a nearly square sheet iron frame, 28 inches high, 16 inches wide, and 14 inches deep, lined with fire brick. The cover is cast iron, as are also the doors to the muffle and ash pit. In the muffle door is a window filled with mica so that the operations going on inside the muffle may be observed when the door is closed. The ash pit doors are provided with wheel openings to further regulate the draft. The grate is formed of cast-iron bars resting upon a cast-iron frame. It is made for a muffle 12 inches long, 6 inches wide and 4 inches high, and weighs 155 lbs. It costs \$20.00 boxed for transportation. This furnace is also made in a larger size, taking a muffle 15 x 9 x 6 inches, weighing 300 pounds and costing \$35.00.

A small furnace called the "Jackass" is made by the Denver Fire Clay Co. This furnace is somewhat similar to Brown's furnace. It is much lighter, however, weighing 100 lbs. It takes the same size muffle and costs about the same.

**Permanent Coal-Fired Furnaces.**—In the laboratories of mines and smelters, where many assays are a part of every day's work, the proper furnaces are those built of brick. One of the best of these is that described by H. W. Parmelee<sup>1</sup> and used in the assay office of Mr. J. I. Brown, Cripple Creek, Colo., by whom it was designed. The construction of the furnace is evident from Fig. 93, and its good points are thus summed up by Mr. Parmelee:

First, it has larger capacity than the ordinary furnace, being capable of being built for either four or five muffles. This enables the operator to conduct several different operations in the furnace at the same time, thus greatly hastening the completion of the day's work. Fusion, scorification and cupellation can be carried on at the same time, and by constantly advancing the

<sup>1</sup>Western Chemist and Metallurgist, III. (1906), 179.

assay through the necessary stages, it is possible to run through a large number of samples. Fusions are made in the lower and upper muffles directly over the fire box; scorifications in the next right-hand muffle and cupellations in the last. The theoretical capacity of this four-muffle furnace is 90 assays per hour. Practically, Mr. Brown has repeatedly handled 400 assays per day of 10 hours, and states that the furnace could easily be made to turn out between 500 and 600 assays per day of 10 hours.

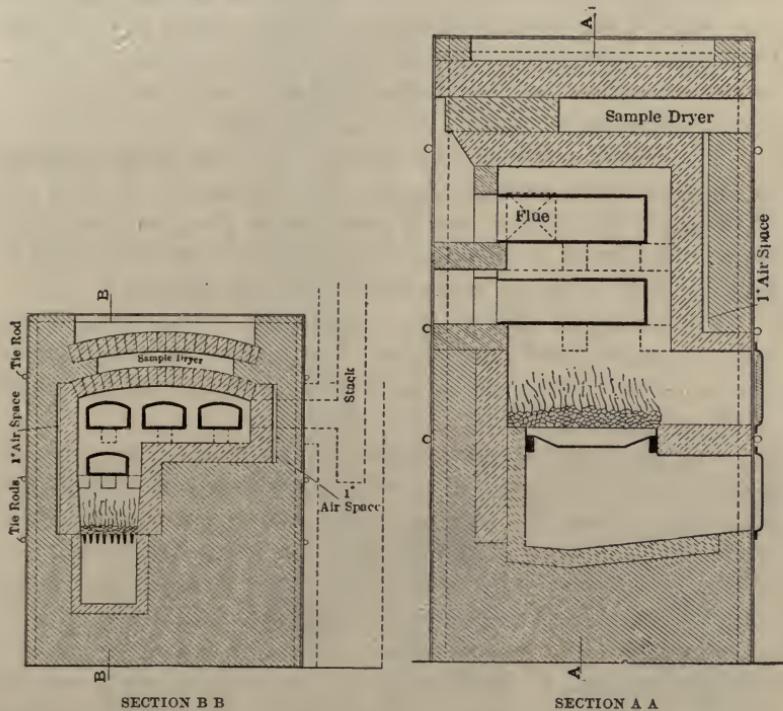


Fig. 93.—Four or Five Muffle Assay Furnace—Mr. J. I. Brown.

Second, it has a sufficiently large combustion chamber to permit of almost complete combustion of the gases before they escape into the flue. This means that the full effect of the heat is received, with the result stated in the preceding paragraph. Five tons of coal per month are required to operate this furnace to its full capacity, running ten hours per day.

Third, it is provided with a sample dryer, which is a valuable adjunct when large numbers of mine samples are to be assayed.

This sample dryer makes use of the heat above the muffles, and has greater capacity than a hot plate or similar contrivance, at the same time being absolutely inexpensive.

Fourth, the fire-clay linings are conveniently replaced when burned out. The front of the furnace surrounding the muffles is made of the ordinary moulded blocks found on the market especially for this purpose. These can be removed and the inside of the furnace is then open for the purpose of replacing the burned-out interior layer of fire brick. It will be noticed in this connection that between the pressed-brick body of the furnace and the fire-brick interior there is a 1-inch air space. This permits of the successive expansion and contraction of the fire brick without placing the stress on the outside walls and tie-rods.

Fifth, it is a forced-draft furnace, the blast being supplied by an electrically-operated blower. To prevent the burning out of the grate bars, which would ensue under the great heat generated, the ash-pit is provided with a cement water pit which is kept full with water up to the level of the ash-pit door.

The materials used in the construction of the furnace are those commonly used everywhere. The outside walls are of red-pressed brick. The inner linings are of fire brick, and the lining of the ash pit is of cement."

Fig. 94 shows a superimposed double-muffle assay furnace<sup>1</sup> used by some of the lead-silver mining companies of Washington and Idaho. Two furnaces are built, one on either side of the stack, only one being used at a time, each being large enough to do the entire work of a large mine. The furnace, when once started, is used from day to day until burned out, lasting from 19 to 22 months. The second furnace is then started while the first is being repaired. By building double, one furnace can always be ready. The repairing of a furnace requires the labor of a mason and helper from two to two and a half days. The stack will not require repairing.

The fuel used is soft coal. The consumption ranges from 175 to 250 lbs. per day, and the assays made will range from 75 to 150 of lead and silver. This furnace can be brought to a proper heat for work in 45 min., but two hours are usually taken. The drawing plainly shows all dimensions.

The grate-bars are of cast iron, as well as all doors, which

<sup>1</sup>Ulysses B. Hough, in *Engineering and Mining Journal*, June 15, 1905.

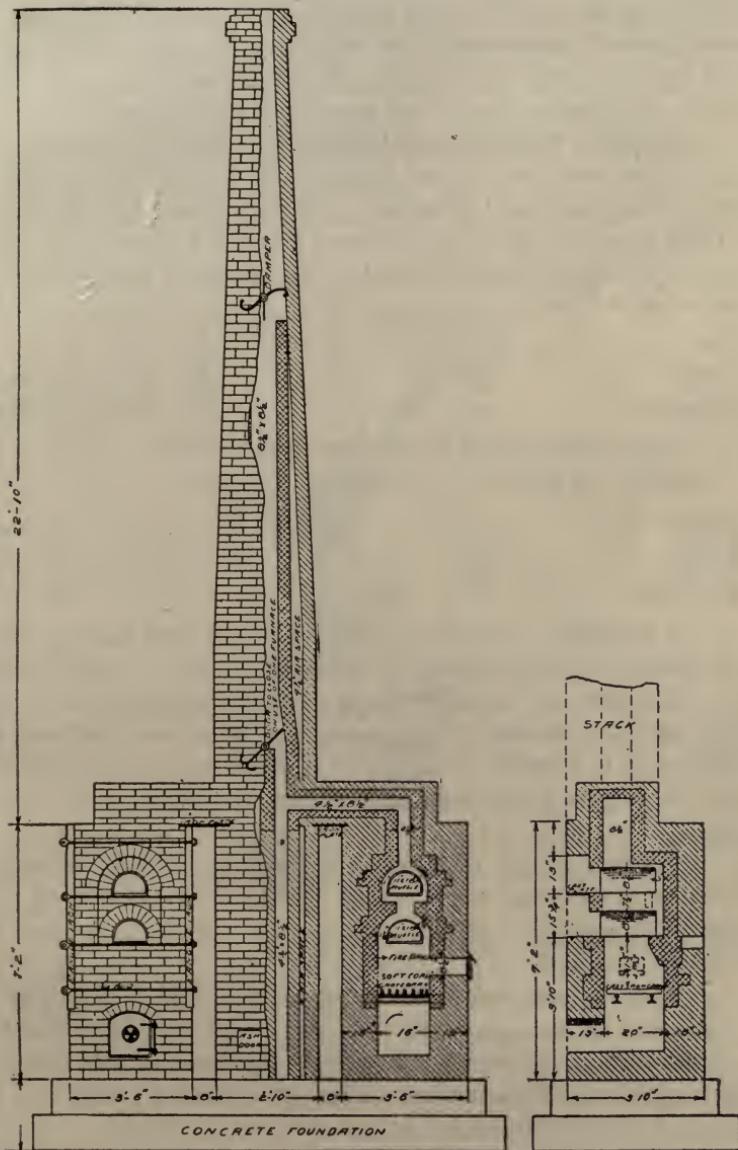


Fig. 94.—Double Muffle Furnace—Mr. Ulysses B. Hough

are lined with cast plates perforated by  $\frac{1}{2}$ -inch holes. The lining only is replaced from time to time. The outside should be securely stayed, as shown. The rods are to be made with a nut in one end and an eye in the other. In this furnace both muffles heat alike, work being done in one as well as in the other.

**Crucibles, Scorifiers and Cupels.**—Crucibles for the assay of gold, silver and lead ores are made of fire clay. They are made in two forms, round and triangular. For an ordinary crucible charge of one assay ton of ore and fluxes a crucible 5 inches high and 3 inches in diameter can be used. The Battersea crucibles go by letters as follows:

Size.....	C	D	E	F	G	H	J
Height.....	3 $\frac{1}{2}$	4	4 $\frac{1}{2}$	5	5 $\frac{5}{8}$	5 $\frac{7}{8}$	6 $\frac{5}{8}$ inches
Diameter.....	2 $\frac{1}{4}$	2 $\frac{3}{8}$	2 $\frac{7}{8}$	3	3 $\frac{3}{8}$	3 $\frac{3}{4}$	4 $\frac{3}{8}$ inches

The crucibles made by the Denver Fire Clay Co. are known as "5 gram," "10 gram," etc., crucibles, as follows:

Capacity.....	5	10	15	20	30	40	grams
Height.....	2 $\frac{5}{8}$	3	3 $\frac{1}{2}$	3 $\frac{3}{4}$	4 $\frac{3}{4}$	5 $\frac{5}{8}$	inches
Diameter.....	2 $\frac{3}{8}$	2 $\frac{5}{8}$	2 $\frac{7}{8}$	3	3 $\frac{1}{4}$	3 $\frac{3}{8}$	inches

A low form 30-gram crucible  $3\frac{3}{8}$  inches high and  $3\frac{1}{2}$  inches in diameter, is also obtainable for fusions made in a small muffle.

Scorifiers are made of fire clay and run in sizes from  $\frac{1}{2}$  inch to 4 inches in diameter. Those  $2\frac{3}{4}$  or 3 inches are most used and are suited to changes from  $1/5$  to  $\frac{1}{2}$  assay ton. For scorification and to reduce a too large lead button the  $2\frac{1}{4}$ -inch size is well suited.

Cupels are made of bone ash and may be made by the assayer himself or they may be purchased. A cupel should be about  $1\frac{1}{3}$  times the weight of the lead button to be cupelled. A cupel  $1\frac{1}{2}$  inches in diameter is a good size.

**Miscellaneous Assay Laboratory Equipment.**—The rest of the equipment of the assay laboratory has much of it been described elsewhere. If the cupels are made by the assayer, a machine for this purpose will be needed. Figs. 95, 96 and 97 show the one made by F. W. Braun & Co., Los Angeles, California.

This machine consists of a hopper to hold the moistened bone ash, and a removable disc with a number of holes which are automatically filled and brought into position under the plunger. To operate, the bone ash is properly moistened and placed in the

hopper. In this latter is a small wheel with a rubber rim that keeps the material stirred up and fills the moulds. The lever handle is then raised and the filled mould brought beneath the plunger by means of the handle on the lower disc. See Fig. 95. The downward motion of the lever handle compresses the cupel (see Fig. 96) and by pulling the disc handle in a reverse direction to that formerly given it, the opening in the lower disc is brought beneath the cupel, and further pressure on the lever handle brings a new system of levers into action, expelling the cupel; which may be caught in the hand (see Fig. 97). An automatic attachment stops the discs at the proper points, and an adjusting device is arranged for giving different degrees of compression.

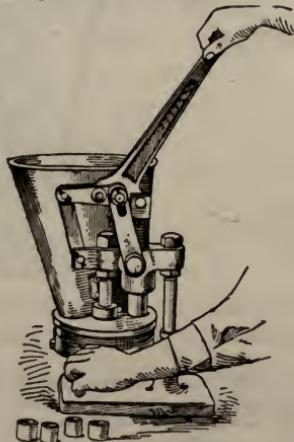


Fig. 95.—Braun's Cupel Machine—Filling the Mould.

Cupels of five various sizes and depths may be made by using interchangeable discs and dies, which are easily adjusted to the machine.

Its list price is \$37.50 fitted for making one size cupels. Dies and discs for other sizes are \$10.00 per size.

The bone-ash, which can be purchased in bulk, is moistened with sufficient of a strong solution of carbonate of potash in warm water to make it of about the consistency of damp sand. It should not be pasty, but should show the finger prints, and adhere well together when squeezed in the hand. The cupels should be dried slowly to avoid cracking.

For removing the cupels, scorifiers and crucibles from the furnace, tongs will be needed. These are made in a variety of

forms which are illustrated in catalogues of assay supplies. For lifting crucibles, when the operator is above them a pair of "basket" tongs are best suited, while for removing them from a muffle a pair of double bent tongs are best. Cupped lip, or lap lipped cupel tongs grip the cupel most firmly; the former will allow the moving of the cupel from the back to the front of the furnace, and when the muffle is full of cupels. Both the cup and lap-lipped tongs grip the cupel much tighter than the plain lipped. Judson has devised tongs for use with both cupels and scorifiers

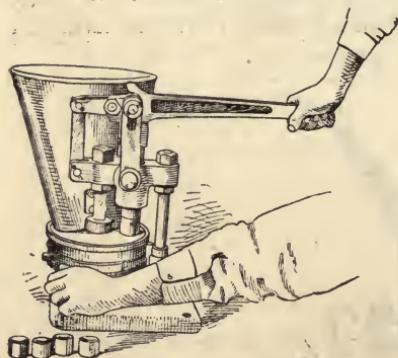


Fig. 96.—Braun's Cupel Machine—  
Compressing the Cupel.



Fig. 97.—Braun's Cupel Machine  
—Expelling the Cupel.

which are convenient. They are described in Brown's Manual of Assaying and listed by Eimer & Amend.

Scorification moulds are also used into which the contents of scorifiers are poured in order that they may cool quickly. They are made of iron and are illustrated in all catalogues of assay appliances.

A muffle scraper is useful for scraping out the muffle after a spill. This consists of a piece of  $1/16$ -inch sheet iron,  $6 \times 6$  inches, bent at right angles, one inch from the end and riveted to an iron rod  $1/4$  inch in diameter and 3 ft. long.

## CHAPTER XIV.

### MISCELLANEOUS LABORATORY EQUIPMENT.

**Aspirators.**—The simplest form of aspirator consists of two large bottles, *a*, Fig. 98, both of which are tightly stoppered with rubber stoppers. Through each of these stoppers pass two tubes, one reaching from a few inches outside to within a fraction of an inch of the bottom of the bottle, the other reaching to just inside the stopper. The two long tubes are joined by rubber tubing and the flow of water through this is regulated by a Hoffman clamp on the tube. If air is to be sucked, the short tube of the upper bottle is to be connected with the apparatus from which the air is to be drawn. If air is to be forced through something, connectio~~n~~ made between the latter and the short tube of the lower bottle.

An improvement on the above is a pair of regular aspirator bottles. These consist of bottles having a tubulature, or opening, at the bottom, *b*, Fig. 98. This is to be closed by a stopper having a short piece of glass tubing, over which is slipped the rubber tube connecting the two bottles. A better form of aspirator bottle has the tubulature drawn out so that the rubber tubing may be attached directly over this, *c*, Fig. 98.

An aspirator bottle may be made by boring a hole in an acid bottle, near the bottom, with a file dipped in turpentine, and then slipping into this hole a bit of glass tube covered with about an inch or so of soft thick walled rubber tubing.

Aspirators made of zinc, japanned and mounted so that continuous suction can be obtained for a long time without changing the connections, may be purchased. The small filter pumps described in Chapter III. may be used for suction, or the blast described in Chapter VI. for forcing air through an apparatus. If the pressure created by the latter is too great, the little regulator described below may be used. This, Fig. 99, consists of a cylinder or bottle stoppered with a 2-hole cork or rubber stopper. One of the holes is left open and through the other passes a T-tube,

one end of which dips into a little mercury in the bottle. The pressure of the gas is regulated by the distance into the mercury which the T-tube dips. Rubber tubes on which are Hoffman

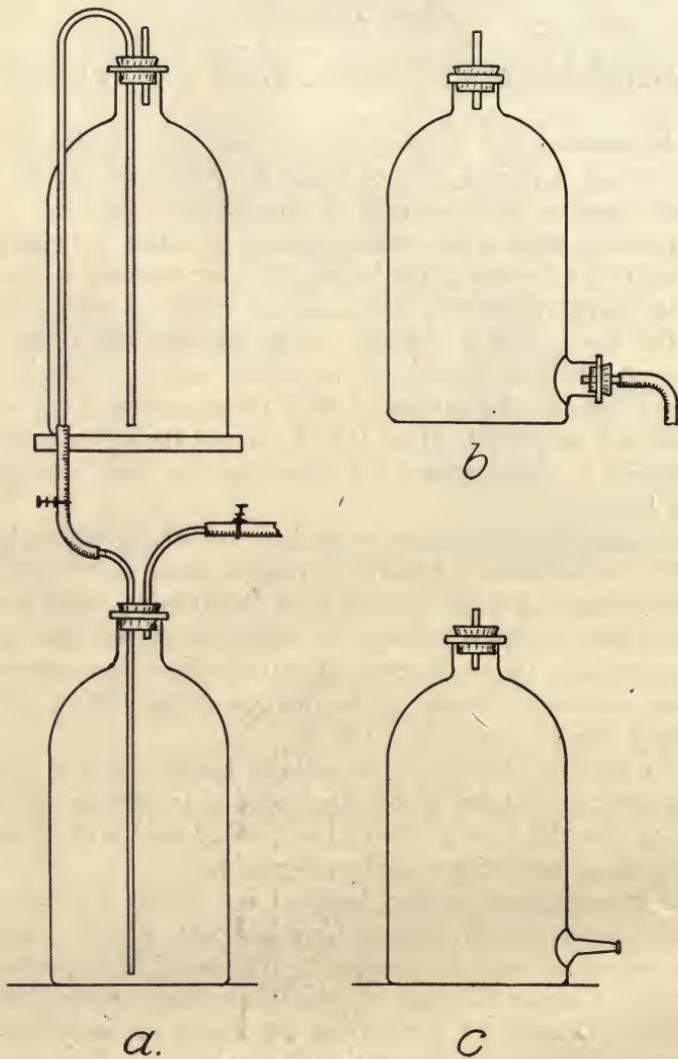


Fig. 98.—Aspirators.

clamps should be used to connect the upper branches of the T-tube, one with the blower and the other with the apparatus through which air is to be forced.

**Barometers.**—These may be obtained in a variety of forms, costing from \$10.00 up. The simple Bunsen's siphon barometer, consisting of a tube of mercury, the upper end of which is closed and the lower is bent to form a U and left open, is well suited to laboratory purposes, for gas analysis, etc. The tube is graduated into millimeters and is mounted on a board, so that it may be conveniently hung up. These siphon barometers may be obtained either filled with mercury or unfilled. If the former they are liable to break in transit, but they are also very troublesome to fill. Aneroid barometers are unsuited to laboratory use. A barometer to be used for gas analyses, etc., should preferably be graduated into millimeters, as nearly all tables, etc., are constructed to use this unit pressure.

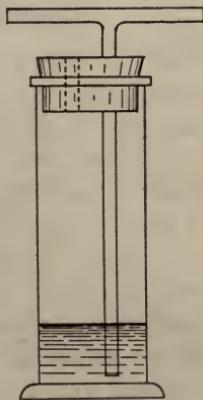


Fig. 99.—Pressure regulator.

**Beakers.**—The best beakers are made of what is known as "trade-mark glass." That is, the maker's name is indelibly stamped upon them. For analytical purposes, the lipped beakers are to be preferred and for most determinations, the Griffin's, or low form, is used. Tall beakers are useful for hydrogen sulphide precipitations. Of the trade-mark glasses the "Jena" comes from Schott and Gen. Jena; the "Nonsol" from Whitall, Tatum & Co., Philadelphia; the "Baloc" from Bausch & Lomb Optical Co., and the "Kavalier" from Joseph Kavalier. "Bohemian Glass" really means very little and tests have shown it to be no better than some of our less pretentious domestic glasses.

Unfortunately dealers in chemical glassware do not adopt a uniform system of numbering their beakers. Herewith will be

found a table giving the number and capacities in cubic centimeters of various makes of Griffin's form beakers:

Make.	No.	0000	000	00	0	I	2	3	4	5
		cc.	cc.	cc.	cc.	cc.	cc.	cc.	cc.	cc.
Baloc .....		20	50	100	150	200	350	500	750	
Jena .....			50	100	150	250	400	600	800	
Kavalier .....		15	30	50	75	120	200	300	450	
Nonsol <sup>1</sup> .....	30	60	75	120	180	250	400	600	750	
		6	7	8	9	10	11	12		
		cc.	cc.	cc.	cc.	cc.	cc.	cc.		
Baloc .....	1000	1500	200	2500	3000	4000	5000			
Jena.....	1000	1300								
Kavalier ....	650	900	1100	1800	2400	3000	4000			
Nonsol.....	1000									

**Carboys.**—These are used in laboratories for the storage of solutions, and acids, ammonia, etc., are received in them. The problem with them is usually how to get the contents out of them. One of the easiest ways is to place the carboy in a Stevenson's tilter. This consists of a pair of rockers so fixed that the carboy can be readily tilted on its side. They can be easily attached and removed from the carboy. Another simple way of drawing from a carboy is to set the carboy on a brick or block a foot or so from the floor and draw the contents by means of a siphon. A pump may be purchased or made for emptying carboys, consisting of a large rubber stopper perforated with two holes, through one of which passes a tube long enough to reach to the bottom of the carboy and having its upper end bent into a wide U, so as to deliver the liquid into a bottle or beaker. Through the other hole of the stopper a short piece of tubing reaches just inside and is connected with an ordinary bicycle foot pump. The rubber stopper is now forced into the mouth of the carboy and wired or tied securely in. The acid is then forced out by working the pump. The bought apparatus has a special form of clamp to keep the stopper in the bottle when the pressure in the latter gets up to the point of forcing the acid out.

For agitating the contents of the carboy, as in making solutions in large quantities, blowing compressed air through the liquid by means of a long tube reaching to the bottom of the

<sup>1</sup>Nonsol Beakers No. 0 A contain 150 cc., 2 A, 300 cc. and 3 A, 500 cc.

carboy will prove convenient and efficient. Where air would oxidize, carbon dioxide or hydrogen may be generated in stone jugs, washed and passed through.

**Casseroles.**—These are nothing more than evaporating dishes with flat bottoms and handles to the side. They are made of both Royal Berlin and cheaper porcelains, and in sizes to contain from 30 to 2000 cc. They are usually used with porcelain handles, though some forms have wooden handles and are also provided with covers. Porcelain casseroles are much used in iron and steel laboratories. They are open to the same objections as are held against porcelain dishes (see dishes). They are much less fragile than beakers and hence are well suited to the rough usage of

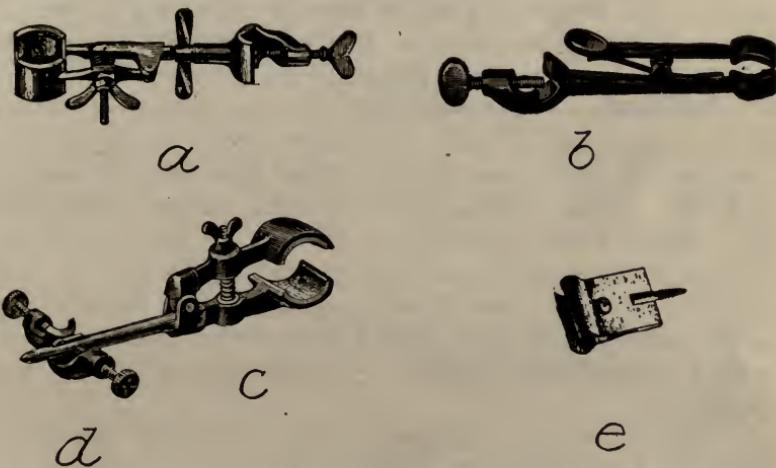


Fig. 100.—Clamps.

laboratories where many determinations are made in a short time. They may be removed from the hot plate or sand bath after the handles become hot by slipping over the latter, just before removal, a piece of large bore rubber tubing, large enough to go on and off without friction. Precipitates may be rubbed out of them easiest with a rubber finger cot drawn over the fore-finger.

**Clamps.**—These are shown in Fig. 100. Forms *a* and *b* are used for burettes and other thin tubular apparatus. Form *a* will hold the burette only parallel with the rod of the support. The jaws being closed with a spring, it is an easy matter to unclamp anything from them. The form *b* will hold the tube at any angle to the rod of the support, and is an excellent clamp for general

laboratory use, for this reason. Clamp *e* is intended to screw into a wall or desk and is so constructed that the graduated part of the burette is not covered. All of the above clamps hold the burette or tube at a fixed distance from the rod of the support. The clamp *c* will allow the rod to be held at any distance up to 5 or 6 inches. It is attached to the support rod by the clamp *d*. This latter clamp may be obtained with a swivel in the middle, controlled by a thumb nut and with this the clamp may be attached to the rod at any angle. Large clamps, similar to *c*, with grips adaptable to irregular shapes, may be obtained for holding tubes over one inch in diameter.

If the jaws of the clamps are not provided with cork grips, a piece of felt should be glued in them in order to give them a securer hold on glass apparatus. Hoffman clamps and pinch cocks are described under rubber tubing.

**Condensers.**—Every catalogue of chemical apparatus will describe many forms of these. Condensers which are intended to condense the vapors and drop the liquid back into the flask in which the boiling is taking place are called "reflex" condensers. The simplest form of condenser is Liebig's. *a*, Fig. 101, shows a simple Liebig's condenser which may be made by any one who is at all proficient in glass blowing, and *b*, Fig. 101, shows a condenser which may be made from a large piece of glass tubing or even a student's lamp chimney. The construction of both are evident.

The end of the condenser in the boiling flask should be ground to a V-shaped point, as shown in Fig 101, in order to facilitate the return of the liquid to the flask or retort. The cold water should always enter at the end of the condenser furthest from the retort. In starting a reflex condenser, it may be necessary to reverse this arrangement, for a second, by turning the condenser upside down, in order to work out the air.

**Dishes.**—These may be obtained from dealers in chemical apparatus, made of porcelain, glass, platinum, nickel, iron, silver, lead, copper, etc. For analytical purposes dishes of porcelain and platinum are usually used and sometimes dishes of glass and silver.

Porcelain dishes are much used in analytical laboratories for silica determinations. In spite of this fact, they are untrustworthy, owing to the fact that it is almost an impossibility to remove all

the silica from them. When silica is determined in steel and iron by Drown's method, they probably give good results, but for silica in slag, clay, etc., there is usually a milligram or so which no amount of rubbing will remove from the dish. The first cost of platinum dishes is, of course, high, but all the silica can be removed from them and they are practically indestructible, so that the increased accuracy and the breakage of porcelain dishes very soon amounts to more than the interest and depreciation on the platinum ones. I have used dishes of Nonsol glass and found them preferable to porcelain dishes for silicate work. If platinum is used, however, the work is unquestionably more accurate, and

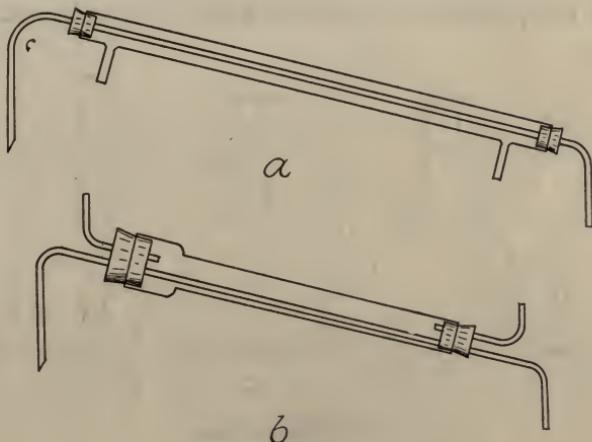


Fig. 101.—Condensers.

owing to the fact that metal is a good conductor of heat, solutions in them evaporate much faster.

To clean precipitates out of dishes, a rubber finger cot will be found convenient. This is slipped over the index finger and the dish rubbed hard. If glass dishes are used, the completeness of the removal of silica may be tested by rinsing the dish with hot water, when it will dry almost at once, and the silica may be readily seen and removed by vigorous dry rubbing with a small piece of ashless filter paper. This latter is then incinerated with the main body of the silica.

**Flasks.**—These may be obtained in some fifteen or twenty different forms, of which the Erlenmeyer and the globe are most used in analytical laboratories. The Erlenmeyer is shown in *a*,

Fig. 102. It is an excellent flask in which to filter by suction (see Chapter III.), for titrations, where precipitates are to be formed in a flask by shaking, etc., and from which to filter by decantation. Flasks to be used with suction should be heavy enough to stand heavy pressure. A very heavy Erlenmeyer with a side neck is made especially for this purpose. For most work an Erlenmeyer with a rather wide mouth will be found convenient. These flasks are made as large as 1 gallon and as small as 1 ounce capacity and can be obtained up to 32 ounces with ground glass stoppers, or with a pour out.

Globe flasks, *b*, Fig. 102, are made in sizes ranging from  $\frac{1}{2}$  ounce to 5 gallons, and with round or flat bottoms. When they are to bear corking they should have what is known as ring-

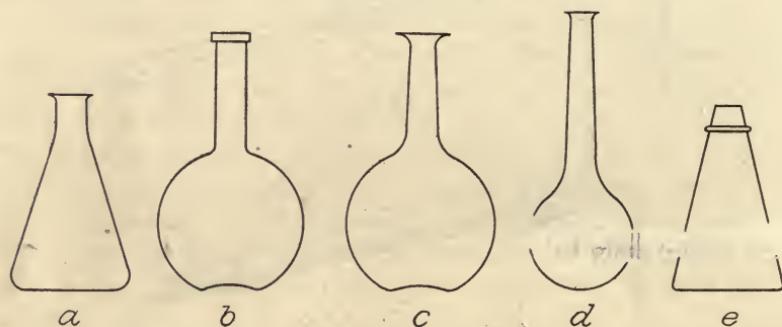


Fig. 102.—Flasks.

finish necks, that is, a heavy ring of glass around the neck, as shown in *b*. The inside edge of the ring should also be ground off to prevent cutting of the stopper. When the flask does not have to be corked a vial mouth may be used, *c*, Fig. 102. For wash bottles (Chapter V.) the ring-finish, flat-bottomed flask should be used.

A small conical flask having a ring around its upper end and known as an "assay flask" or "matrass," *e*, Fig. 102, is used in parting gold bullion. A round bottom flask, *d*, Fig. 102, with a long neck, made of hard Bohemian glass, is used for nitrogen determinations by Kjehldahl's method. Globe flasks, both with flat and round bottoms, having side necks, are used in making boiling point determinations and also for determining substances by evolution and distillation methods.

Any of the foregoing flasks may be obtained made of any glass desired, Kavalier's, Bohemian, Baloc, Nonsol, Jena, etc. (see Beakers).

**Gases, Drying and Absorbing.**—For this purpose U-tubes, potash bulbs, calcium chloride tubes and calcium chloride jars are used. U-tubes, Fig. 103, are made in a number of forms and are suited to drying gases by the use of calcium chloride, anhydrous copper sulphate, sulphuric acid, etc. Figs. *a* 103 and *b* 103 are good forms where the gas has merely to be dried or purified and the form shown in Fig *c* 103, with glass-stoppers, is to be preferred where the gas has to be caught and weighed. The other forms can also be used for this purpose. In this case, the tubes should be capped when they are not connected in the train

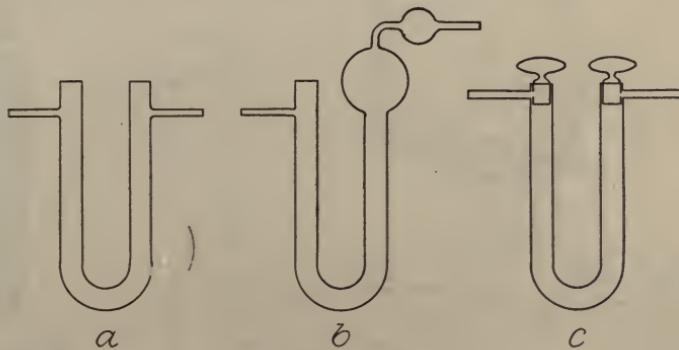


Fig. 103.—U-Tubes.

by means of small pieces of thin walled rubber tubing, one end of which are closed by bits of glass rod or, better still, capillary glass tubing. To suspend tubes to be weighed, use fine platinum wire. U-tubes, in which liquids are to be used, should be filled with glass beads or cracked up pumice-stone, and should contain only sufficient liquid to cover the bend. Where dry substances are used, plugs of loose absorbent cotton should be placed on top of this, in both branches of the tube, to prevent fine particles from being carried out of the tube. Where tubes are stoppered with corks the ends containing these should be dipped in melted paraffin or wax, so that all the cork and some of the end is covered to prevent escape of the gas.

Calcium chloride tubes are shown in *a* and *b*, Fig. 104. They may be obtained either with one or two bulbs. The form shown

in *b*, Fig. 104, has a small tube extending into the bulb and is intended to catch some moisture (as water) by condensation in the first bulb, and so save the calcium chloride. These tubes are seldom weighed and are usually used as guard tubes. The calcium chloride jar is shown in Fig. 104 *c*. It is never weighed and is usually used only to purify or dry air or gases. A small pad of asbestos fibre or cotton is placed at the constricted point just above the tubulation and the purifying substance on this. They may also be used for liquids by filling the upper portion with glass beads and drenching these with the liquid. They may be used for both liquids and solids by having the lower part of the tube filled with liquid nearly to the tubulation and having the tube by which

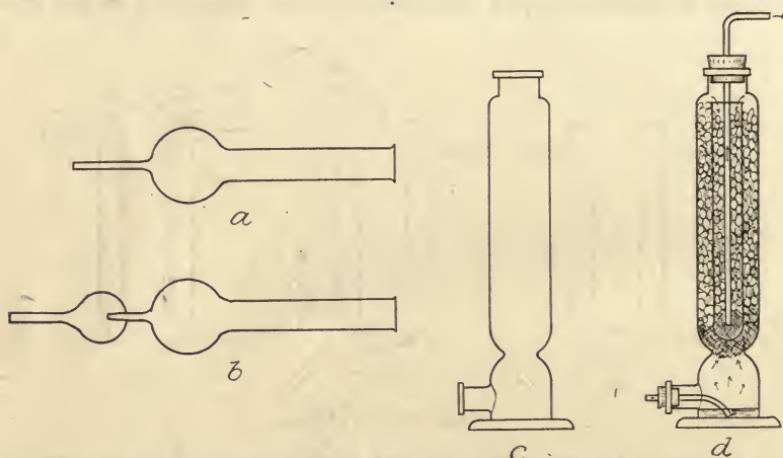


Fig. 104.—Calcium Chloride Tubes and Jars.

the gas enters bent so that the latter will have to bubble through this. Fig. 104, *d*, shows a very powerful purifying apparatus made from a calcium chloride jar and a long narrow test tube. The lower part is filled with sulphuric acid (caustic potash for  $\text{CO}_2$ ) and the upper part and test tube with calcium chloride (soda lime for  $\text{CO}_2$ ). The test tube rests on a plug of loose cotton and the gas is led out of the apparatus by means of the tube as shown. In this apparatus, the gas has to travel the entire length of the jar and also the test tube, as shown by the arrows.

Potash bulbs are for use with liquids only, though there is usually attached to them when they are to be weighed a tube containing calcium chloride or soda lime. The most common forms

are shown in Fig. 105. Where the bulb is to be weighed the form marked *b* is best suited, or the form *a* may be weighed together with a U-tube for a guard. When not connected up in the train they should be capped as directed for U-tubes. To fill the bulbs, attach a short piece of rubber tubing to one end and dip the other in the solution held in a small shallow dish. Apply suction to the rubber tubing with the mouth until the bulbs are filled to the proper height. Then wipe the tube dry inside and out with pieces of filter paper or a soft cloth.

Gas may also be purified by allowing it to bubble through solutions held in 4-ounce bottles.

**Gas Generators.**—There are many forms of these, and to de-

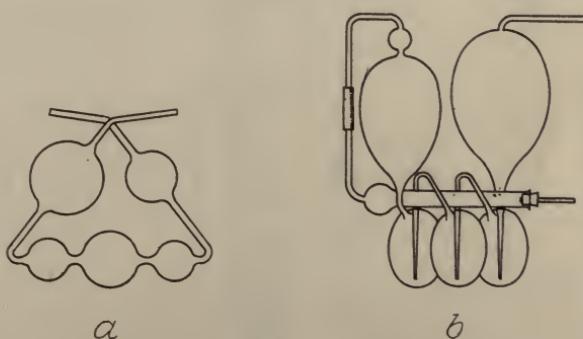


Fig. 105.—Potash Bulbs.

scribe them would of itself fill a volume. A simple generator for hydrogen sulphide, carbon dioxide or hydrogen is shown in Fig. 106. The acid is held in the bottle, *A*, which is provided with a tubulature. The iron sulphide, etc., is in the calcium chloride jar, *B*. The acid flow is regulated by a Mohr's clamp, *a*, and trickling through the sulphide generates the gas. The spent acid falls into the lower part of the jar and is sucked off into a waste bottle by the siphon, *D*. The gas is washed by bubbling through water in *E* and its flow is controlled by the Mohr's clamp, *e*.

**Hydrometers.**—These are used for taking the specific gravity of liquids. Some of them are very accurate, reading to the third and fourth decimal place. For milk, wine, oils, etc., special hydrometers are employed and they will be found listed in the catalogues of the various dealers in chemical supplies. For ordinary

laboratory work two hydrometers will be found useful: One for taking the specific gravity of liquids heavier than water and one for use with those lighter. Since temperature affects specific gravity, hydrometers having thermometers blown in them are to be preferred, as temperature and density can be read at the same time. Universal hydrometers are also made for use with liquids either lighter or heavier than water. Very delicate hydrometers usually have but a small range and hence are usually purchased in sets. Hydrometers reading in degrees Baumé, Brix, Balling,

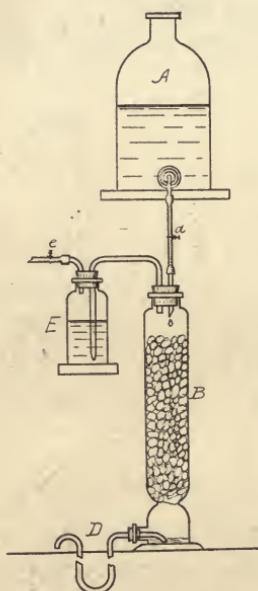


Fig. 106.—Gas Generator.

etc., instead of specific gravity compared with water are also used. Hydrometer jars are tall narrow cylinders, mounted on a foot, in which to float hydrometers. The Westphal Balance is also used to take the specific gravity of liquids and is very convenient and accurate.

For taking the specific gravity of both solids and liquids, small bottles having narrow holes drilled through the stoppers to enable the operator to fill them precisely, are used. They are called specific gravity bottles or pycnometers. Fig. 107 shows

the forms most commonly used, of which *a* and *b* are the cheapest, and *c* is the best, while *d* and *e* (Sprengel's tubes) are used for taking the specific gravity of liquids where only a small amount is available. The tube shown with the latter two forms is used to fill them.

**Measuring Apparatus.**—The chief forms of these are burettes, pipettes, graduated flasks, graduated cylinders and "graduates." Burettes have already been described, and an automatic pipette was also mentioned, both in Chapter VII.

For roughly measuring reagents, graduated cylinders are usually used. These may be obtained in sizes ranging from 5 cc.

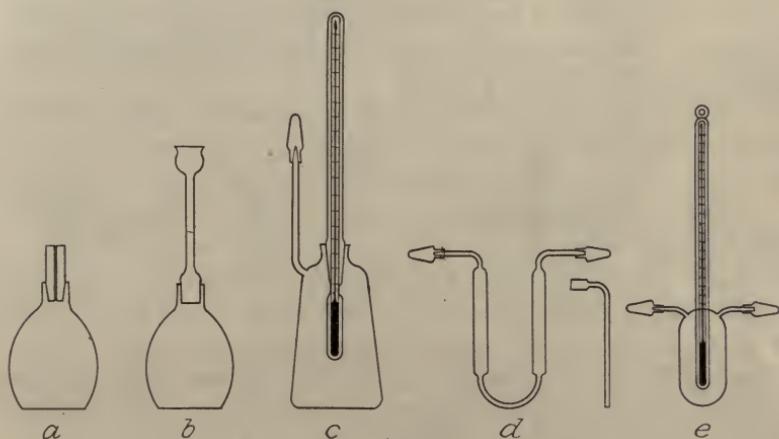


Fig. 107.—Specific Gravity Bottles.

to several liters. A convenient form is manufactured by Whitall, Tatum & Co., in which the foot is made very heavy in order to make the cylinder less liable to turn over. Cylinders can be obtained with double graduations and also with ground glass stoppers. The latter form are handy for making up dilute acids and solutions in which two liquids are used.

For accurately delivering small volumes of solutions, pipettes are used. These are made in sizes ranging from 5 to 100 cc. and are filled, to a given mark on the stem above the bulb, with the liquid by suction and then allowed to empty and drain themselves. Graduated pipettes, which are practically nothing but small

burettes, the flow out of which is controlled by the finger on the upper end, can also be obtained and with these any volume can be delivered. They are made as small as 1 cc., graduated into  $1/100$  cc., and as large as 50 cc., graduated into  $1/10$  cc. Special pipettes are also made. Some bulb pipettes are made to deliver a certain volume between a mark on the upper stem and one on the lower. Pipettes are usually marked to show at which temperature the given volume is delivered.

Graduated flasks or "Volumetric Flasks" are usually made to hold a certain volume when filled to a given mark on the neck, although they can be obtained with two marks, the lower of which denotes the volume contained and the upper the volume delivered. Flasks for volumetric work should preferably have ground glass stoppers. The sizes kept in stock by dealers in chemical glassware are from 25 cc. to 2 liters.

Measuring apparatus may now be obtained accompanied by certificates of the U. S. Bureau of Standards as to correct volume, or it may be sent to them for verification.

**Motors.**—For laboratory purposes electric and water motors will be found most convenient for furnishing power. A small  $\frac{1}{4}$  H.P. motor will run almost any piece of machinery about an analytical laboratory and these may be purchased for between \$40.00 and \$50.00, wound for 110-volt or 220-volt circuit. For running crushing and grinding machinery, a 1 H.P. motor will usually be sufficient and this can be obtained for about \$75.00. For running a few stirrers, rotating anodes, etc., a No. 7 Porter motor wound either for battery (\$7.00) or power (\$8.00) may be used to advantage, a leather shoe-string serving for a belt.

Rabe's small water motors are also useful for running a stirrer or two. They cost \$6.75 with holder to attach them to a support. Connections are made to water faucets with stout rubber tubing. Larger water motors, the power of which will of course depend on the water pressure, are obtainable, in sizes ranging from  $1/10$  to 4 or more horse power. If good water pressure is at hand they are cheaper than electric motors.

Where neither electricity nor gas are obtainable hot air motors may be used. They are very expensive, however, a  $1/5$  H. P. motor costing about \$300.

**Rubber Stoppers.**—These should be of the purest gum and

can be obtained solid or perforated with one, two or even three holes. They are made in the following sizes:

Diameter of Top.		Diameter of Bottom.		Diameter of Top.		Diameter of Bottom.			
Size.	mm.	ins.	mm.	ins.	mm.	ins.	mm.	ins.	
00	14	0.55	10	0.39	7	37	1.46	30	1.18
0	17	0.67	12	0.47	8	41	1.61	33	1.30
1	18	0.71	15	0.59	9	45	1.77	37	1.46
2	20	0.79	16.5	0.65	10	50	1.97	42	1.65
3	23	0.91	18	0.71	11	56	2.20	50	1.97
4	25	0.98	20	0.79	12	65	2.56	59	2.32
5	27	1.06	23	0.91	13	70	2.76	60	2.36
6	32	1.26	26	1.02					

To push glass tubing through the perforations of rubber stoppers, first round the end of the tube with a file, or by heat,

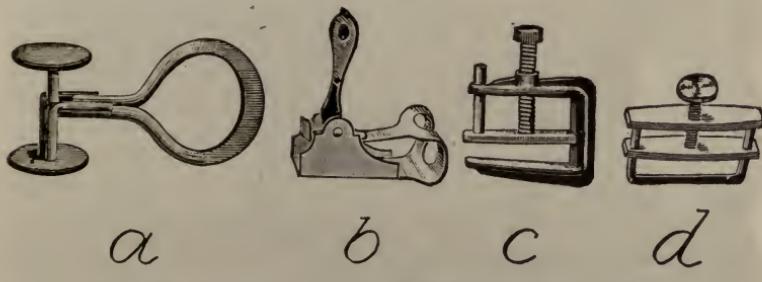


Fig. 10S.—Hoffman's Clamps and Mohr's Pinch Cocks.

and, after cooling, moisten the rod and hole. Water acts as a lubricant between rubber and glass.

**Rubber Tubing.**—Rubber tubing for connecting burners to gas taps should be heavy walled with so-called "cloth impression" tubing of about 6 to 8 mm. ( $\frac{3}{4}$  inch) internal diameter and having walls 3 to 4 mm. thick. Tubing of this size may also be used for filtering by suction.

For connecting up glass apparatus and for general laboratory use, a tubing of pure black or red gum should be used. It should be seamless and may be obtained either with thick or thin walls and in sizes ranging from 3 to 18 mm. internal diameter. In my own laboratory, I use red rubber tubing with thick walls and find 3 mm., 5 mm., 6 mm., and 9 mm. good sizes to keep on hand.

For controlling the flow of liquids and gases through rubber tubing, Hoffman's clamps, *c* and *d*, Fig. 108, and Mohr's pinch cocks, *a* and *b*, Fig. 108, are used. With the former the flow can be regulated, but the latter is intended only to shut off the flow

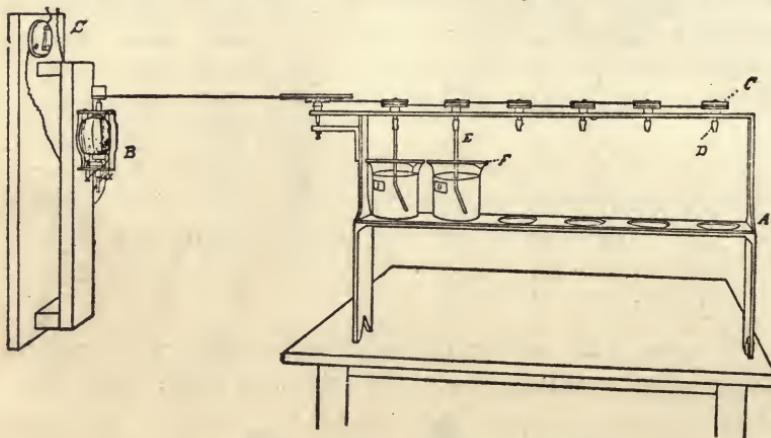


Fig. 109.—Blair's Mechanical Stirrer.

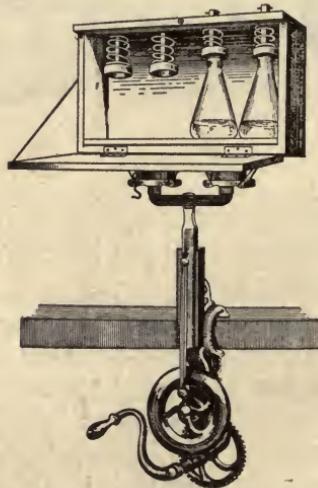


Fig. 110.—Dudley's Mechanical Shaker.

suddenly. Improved forms of Hoffman's clamps are now made which can be attached to tubing without disconnecting the latter, Fig. 108, *c*.

**Stirrers, Shakers, Etc.**—A great variety of mechanical con-

trivances for stirring liquids are on the market, of which Fig. 109 shows one of the simplest, devised by Blair, and illustrated in his "Chemical Analysis of Iron." It may now be purchased of dealers in chemical supplies.

A simple shaker for precipitation is Dudley's, shown in Fig. 110, and this also is a stock article. Numerous forms of shaking devices are illustrated and described in various text books of analytical chemistry.

Mr. Robert Job described<sup>1</sup> a simple method for agitating solutions by means of air blown through them. The air is first



Fig. 111.—Job's Air Agitator.

made to bubble through water to take out the dust, etc., and is then passed into the solution through a glass tube. For dissolving steel in cupric chloride solution, Mr. Job placed the drillings in a test glass. Fig. 111 shows the apparatus. For phosphorous determinations Erlenmeyer flasks were used and for magnesia precipitates the author has used beakers.

**Thermometers.**—For ordinary purposes, chemical thermometers having a long thin stem, about  $\frac{1}{4}$ -inch bore, and the scale engraved on the glass, will answer. They can be obtained read-

<sup>1</sup>Chemical Engineer, II., 353.

ing as high as  $360^{\circ}$  C., graduated into degrees, or reading to  $550^{\circ}$  C. graduated every 5 degrees. For calorimeters and such work more delicate thermometers can be obtained, while for research work in boiling and melting points Beckmann's thermometers are used. Thermometers accompanied by certificates as to their accuracy from the Deutsche Physikalische-Technische Reischsanstalt or the U. S. Bureau of Standards can be obtained at a slightly increased cost.

Pyrometers are used for temperatures higher than  $550^{\circ}$  C. Of these Le Chatelier's, Wanner's, the Féry and Bristol's are all good forms, each one of which is best suited to some particular kind of work. They are fully described in various works on metallurgy, thermochemistry, etc.



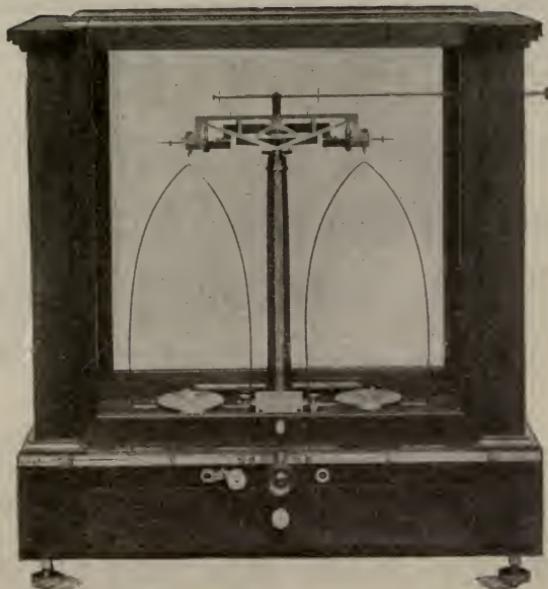
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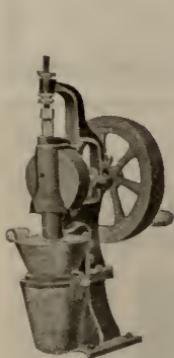
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